METALLURGIA

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Still Need for Economy

JUST over a year ago—in August, 1951, to be exact—the then Minister of Supply, acting in conjunction with the Lord Privy Seal in his capacity as Minister of Materials, set up the Metals Economy Advisory Committee under the Chairmanship of Mr. D. A. Oliver, who had previously been appointed Metals Economy Adviser to the Minister of Supply.

We take this opportunity of extending to Readers our Cordial Greetings for the Festive Season and Good Wishes for the Coming Year

During the last 12 months there have been considerable changes in the metal supply position, and although it cannot be regarded as completely satisfactory, at least the critical shortages have eased somewhat. The Committee has decided, therefore, that the time is opportune for the publication of the first report* on its work. The report reviews the work done by Governmental and industrial organisations to promote metals economy and pays tribute to research associations and private firms. Industry, it says, has been fully alive to the importance of economy and has done much to reduce its requirements of scarce metals by redesigning products, substituting more readily available materials and by other methods, and it is emphasised that it is through the efforts of industry itself that the greatest economies can be made.

A particularly striking saving of tungsten has been achieved by modification of the high nickel-chromium steel used for heat-resisting rings in jet engines, whilst further savings have resulted from fostering the use of ferritic gas turbine discs in place of the more highly alloyed ones, which also reduces the cost and doubles the production owing to the easier manipulation. Other economies on the ferrous side include those made by reducing the nickel and molybdenum contents of certain low-alloy direct hardening and case hardening steels. An interesting possibility is opened up by work at the N.P.L. which has shown that, in steels of particularly low phosphorus content, the alloy content required to attain a given standard of mechanical performance may be lower than the amount required when more phosphorus is present. Progress has also been made in the reduction in tungsten content of high speed steel permitted by an increase in its content of vanadium, an alloying element which has possibilities in other directions. The supply position is reviewed elsewhere in this issue, and methods of increasing supplies of vanadium from indigenous sources suggested.

Considerable attention has been given to economy in the use of 70 : 30 cupro-nickel tubes for steam condensers, and this material is now only specified for use with cooling waters so contaminated that the only tried alternative—aluminium brass—is unsuitable. At the same time, other alternative materials are being studied, and encouraging laboratory tests have been carried out on a copper-based alloy containing 10% nickel and 1·5% iron. Tests under service conditions have been arranged for this material and a 12% alpha tin bronze alloy. The use of aluminium for overhead cables is already well established, but there are a number of difficulties with their use underground, and until the results on trial lengths are known there is little likelihood of their extended use.

Taking the long view, a significant contribution to metal economy can be achieved by extending the service life of metals, and in this connection a considerable body of work is being devoted to the study of the corrosion of metals when immersed in liquids, when buried, and when exposed to atmosphere. Protective treatments play an important part in the struggle against corrosion, and although the coatings themselves may comprise scarce materials, the overall saving may justify their use. Economy has been effected by reducing coating thicknesses to the minimum consistent with satisfactory service behaviour. Too great a reduction can be false economy, but it has been shown that, in certain circumstances, savings of up to 25% can be effected in commercial galvanising plants without detriment to the product.

The expansion of world production and the operations of the International Materials Conference have resulted in bigger supplies of alloying and non-ferrous metals to the U.K. in 1952 than had been forecast. At the same time, demands have been less than expected, and industry's requirements have been restricted by voluntary and enforced economies. There is, however, little margin in estimated supplies to meet unforeseen demands or to build up stocks, and if the rise in production in the metal consuming industries is greater than has been estimated, the situation will certainly not be eased. It will be seen, therefore, that there is every reason for continuing and extending economy measures.

The difficulties of the Committee in promoting a live interest in metals economy are increased now that the metal supply position is apparently easier. The adoption of major economy measures frequently entails expenditure on plant conversion and some short-term disadvantages, and firms are naturally hesitant to commit themselves to projects which may appear to be financially unjustified. For this reason the Government should, through the Committee, provide industry at regular intervals with as much information as is practicable regarding future supplies of the main metals and possible shortages. This, together with the maintenance of a steady pressure to adopt the latest and best techniques ever more widely throughout industry, is probably the greatest service the Committee can perform.

* Report of the Metals Economy Advisory Committee. H.M.S.O. 9d. net.

Vanadium—The Possibility of its Production from Indigenous Sources

By J. Sandor, D.I.C., L.I.M.*

The mineral resources of the sterling area are wholly inadequate to meet the increasing vanadium needs of this country and Western Europe. Most British and some imported iron ores contain a small amount of vanadium, which tends to be concentrated in the steelmaking slag. In this paper, proposals are put forward for carrying out the iron and steel making operations in such a way as to make the extraction of the vanadium from the slag an economic proposition.

VANADIUM is increasing in importance as a supplement to, or even an alternative for, tungsten and molybdenum in high speed steels, in structural steels and in hard metal carbides. Although the uses of vanadium are increasing, high-grade sources of this valuable alloying element are inadequate to meet all requirements. Of the low-grade sources, the most important is the occurrence of vanadium in certain types of iron ore. In iron and steel making practice a build-up of vanadium takes place, and eventually the vanadium finds its way into the steel slag. This paper reviews the various processes by which the vanadium in the ore can be conserved and recovered economically without undue interference with steelmaking practice.

Sources of Vanadium

(a) Vanadium Minerals.

High grade vanadium minerals are not abundant in nature. They occur chiefly together with lead and zinc minerals, and they also accompany certain uranium minerals in the United States and Eastern Europe. The most important producers of vanadium ores are: South West Africa, where vanadium occurs chiefly as vanadinite, a chloro-vanadate of lead, and descloizite, a chloro-vanadate of zinc and lead; Northern Rhodesia (vanadinite); United States, where it occurs mainly as carnotite, which is essentially a uranium mineral; and Peru, where it occurs mainly as patronite, a sulphide of vanadium associated with asphaltic material. Table I shows the production of vanadium minerals, in terms of metal, by the main producing countries between 1938 and 1951.

TABLE I.—PRODUCTION OF VANADIUM ORES.
(In terms of metal—long tons)

	1938	1944	1945	1946	1947	1948	1949	1950
Northern Rhodesia ..	368	251	216	67	56	171	151	—
South West Africa ..	549	279	435	457	222	180	160	290
United States	720	1,575	1,323	568	946	(a)	(a)	(a)
Peru	813	513	661	317	428	503	234	(a)

(a) Figures not available. Source: Imperial Institute.

(b) The Occurrence of Vanadium in Iron Ores.

Vanadium occurs in small quantities in certain iron ores. Of these, the titaniferous iron ores contain the highest percentage of vanadium (0·2–0·8% of vanadium or sometimes even higher values), but they are at present of little industrial value because their high titanium content makes it impossible to work them in an ordinary blast furnace. Magnetites carry up to 0·1% of vanadium. A general, though possibly fortuitous, rule is that the low-phosphorus ores are free from vanadium, whereas the phosphoric ores used in basic steel manufacture usually contain 0·02–0·1% of vanadium.

Although the vanadium content of most iron ores is comparatively small, in iron production the vanadium passes entirely into the pig iron and, in the process, undergoes a two- or three-fold concentration. A further concentration occurs when the vanadium passes into the steel slag, either in the Bessemer converter or in the basic open-hearth furnace.

Due to this process of concentration, the recovery of vanadium from iron ores, on an industrial scale, was practised in France as long ago as 1870, but was soon abandoned, because the demand for vanadium dropped. In Germany, the process was developed to a fine art and during the second world war as much as 3,600 tons of ferro-vanadium were produced annually, using vanadium recovered from iron ores. This not only made the Germans entirely independent of overseas supplies, it permitted a vast programme to be carried through, using vanadium as an alternative to molybdenum in alloy steels and for the partial replacement of tungsten in high speed steels and tool steels.

(c) Other Low-grade Sources of Vanadium.

Small quantities of vanadium can be extracted from flue dust and soot obtained from firing vanadium-containing fuel oils. Before and during the second world war, vanadium-containing soot was collected regularly from British shipyards. This source, however, depends on burning Gulf of Mexico oil, because oil from the Middle East contains very little vanadium. Even when comparatively high vanadium-containing oils are burnt, the soot or flue dust collected does not usually contain more than about 3 to 4% of vanadium. In America, vanadium has been extracted regularly from flue dust, but even there the quantities so obtained have been comparatively small; for example, in 1947, 32 tons of vanadium were extracted in this way, compared with 1,400 tons of vanadium obtained from the ore.

A more important source is oil-refinery residues. Such residues have been imported regularly from Curacao and they contain up to 33% of vanadium pentoxide (18·5%V).

Small quantities of vanadium can be recovered from chrome ores. In America, the vanadium-oxide content of certain chrome ores used in bichromate manufacture averages approximately 0·50% (0·3%V) and efforts have recently been made to recover it on a commercial

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scale[1]. The chrome ore imported to the U.K. contains approximately 0·1–0·2% of vanadium; total imports of chemical chrome ore (mainly from Transvaal and S. Rhodesia) amounted to 35,000 tons in 1951; thus, assuming a 50% recovery, this process would yield vanadium oxide corresponding to about 18 to 35 tons of vanadium per annum.

About one third of the vanadium in bauxite may be recovered from the Bayer process liquors. Such recovery is considered economic if the aluminate liquor contains a minimum of 0·5 g./lit. of vanadium pentoxide. In the U.K., bauxite from the two main sources (France and Gold Coast) contains too little vanadium to be of interest.

Sources of Vanadium at Present Available to the United Kingdom

In the United Kingdom, vanadium is not at present recovered from iron ores. The source from flue dust and soot, though it was important during the second world war, is no longer available, owing to the low vanadium content of Middle East oils, and the difficulties of collection. Oil-refinery residues from Curaçao are still being imported and in 1950 and 1951 they contributed 100 tons of vanadium per annum, i.e., about one third of the total U.K production. This supply, however, is dwindling owing to the lower vanadium-content of the new oil-wells in the area, and much less vanadium is expected from this source in 1953.

Although there are extensive vanadium-bearing deposits in the United States, considerable quantities of ore are imported from Peru. There is, therefore, no known ore containing 6% of vanadium or above, immediately available to the United Kingdom, other than that from Northern Rhodesia and South West Africa. The latter is the more important source; it also supplies most of the needs of Europe.

(*a*) *Northern Rhodesia.*—This is mainly a lead-zinc producing mine. The vanadium in the ores occurs in varying quantities, and has to be extracted by leaching. Since 1950, no vanadium pentoxide has been produced, as the leaching plant has been used for the recovery of zinc and vanadium from fine mixing tails.

(*b*) *South West Africa.*—Mining is carried out under extremely difficult conditions underground. There are difficulties such as shortage of rolling stock, port facilities and shipping. The South West African material reaches this country mainly in the form of residue known as vanadium slag, after the lead has been extracted from the concentrate.

Use of Vanadium

The main industrial source of vanadium is vanadium pentoxide. Approximately 90% of all vanadium pentoxide is smelted with iron to ferro-vanadium, either by the alumino-thermic process, or by reduction with silicon or carbon in the electric furnace; 96% purity vanadium can be made by the alumino-thermic reduction of vanadium pentoxide, and 99·8% purity vanadium (which is ductile)[2,3] can be made by reduction with calcium metal. Vanadium metal has also been prepared by the electrolysis of sodium vanadate in hydrochloric acid.

Table II shows the consumption of vanadium in steels in the U.K. and in the U.S.A. between 1940 and 1948. The main use of ferro-vanadium is in high-speed steels, but important uses include the steels employed at elevated temperatures. On the Continent, considerable quantities of ferro-vanadium are used in low-alloy steels with a vanadium content of approximately 0·2%. The vanadium content of high-speed steels in the U.K. at present is approximately 1–1·5%; though some new high-speed steels now contain 3·0% of vanadium. This at present is possible because only a small proportion is made with this content. In Germany, high-speed steels with 2·5–3% of vanadium gave excellent service during the war, and enabled substantial reduction in tungsten content to be effected.

TABLE II.—CONSUMPTION OF VANADIUM METAL IN STEEL
(Long tons)

	1940	1941	1942	1943	1944	1945	1946	1947	1948
U.K. ..	440	530	535	450	275	210	183	200	191
U.S.A.	(a)	2,024	2,898	3,257	2,427	2,411	1,030	1,434	(a)

(a) Figures not available. Source: British Iron and Steel Federation.

Vanadium has been used as an alloying element in light metals. Small quantities, i.e., 0·1–0·5% of vanadium, have been used to improve impact strength, ductility and grain size in aluminium alloys.

In the chemical industry, vanadium, vanadium oxide and vanadium salts are used chiefly as catalysts. Some vanadium salts have also been used as mordants for dyes.

Vanadium linoleate has been employed as a paint dryer, its action being more rapid than the corresponding lead and manganese salts.

Vanadium in the oxide form, or as ammonium metavanadate, is used in the manufacture of glass and ceramic glazes and in fungicides and insecticides[3].

Alloys of titanium and vanadium are under study at present for elevated temperature application in jet aircraft and guided missiles. Such alloys have shown great promise where high creep resistance at temperature is required[4].

Principles of Vanadium Recovery from Iron Ores

(*a*) *Vanadium-containing Pig Iron from the Blast Furnace.*

In normal blast furnace practice, vanadium goes into the pig iron together with phosphorus and silicon. Titanium on the other hand, goes into the blast furnace slag.

Owing to the fact that vanadium goes from the ore into the pig iron, the important factor, which has often been overlooked when considering the suitability of certain ores, is the V/Fe ratio and not just the absolute vanadium content of the ore. Important data on the vanadium content of British and imported iron ores has been collected and published by T. Deans[5]; some of the figures shown in Tables III and IV have been taken from this paper. If the V/Fe percentage of vanadium-containing British iron ores is compared with those in Luxembourg and Germany from which vanadium has been successfully recovered, it is clear that the British ores are superior to the Minette ores from Luxembourg, though not so good as the Salzgitter ore.

On the basis of vanadium to iron ratio, the imported Swedish ores in general fall into the same category as the British ores, although their total vanadium content is much higher. Low-phosphorus iron ores imported from Spain and Spanish Morocco, on the other hand, do not contain sufficient vanadium and they have not been listed in these tables.

TABLE III.—ANALYSES OF LUXEMBOURG, SALZGITTER AND SOME TYPICAL BRITISH VANADIUM-CONTAINING IRON ORES AND A TITANIFEROUS ORE FROM TRANSVAAL.
(Weight % dry basis)

	SiO_2%	Al_2O_3%	Fe_2O_3%	FeO%	Total Fe %	MnO%	CaO%	MgO%	P_2O_5%	S%	Ti%	Loss %	V%	V/Fe%
Minette (Eitelberg, Luxembourg)	18	5·57			35·5	0·39 (Mn)	14·4	1·1	0·77 (P)			24·23	0·039	0·11
Salzgitter	26·67	9·54			23·17	0·15 (Mn)	5·46	1·51	0·36 (P)			33·05	0·085	0·37
Northampton (Irthlingborough, Sinter)	19·30	8·68	36·75	22·32	43·04	0·34	4·30	1·94	1·78	0·24		3·54	0·078	0·18
Frodingham, Lincs. (No. 3 Face)	8·55	5·18	24·45	9·60	24·54	0·91	21·70	1·81	0·90	0·38		26·07	0·045	0·18
Frodingham, Lincs. Crosby ..	6·75	4·68	24·74	9·47	24·66	1·37	23·00	1·74	1·03	0·38		26·56	0·051	0·21
Frodingham, Lincs. Conesby ..	5·10	4·18	30·53	6·98	26·77	1·37	22·60	1·30	0·76	0·35		26·48	0·046	0·17
Transvaal (Titaniferous)	0·5–3·0		65–74 (Fe_3O_4)	3–9	53–56	Cr 0·1–2·7	0–0·8	0·1–1·25	traces		6·5–7·5		0·4–0·7	0·7–1·3

(b) *Vanadium Pig Iron from the Electric Smelting Furnace*

In regions where there is sufficient cheap coal and cooling water, or where there is cheap hydro-electric power, the smelting of iron ores in electric blast furnaces might be considered. The importance of electric smelting lies in the fact that, up to the present, this is the only industrial process by which titaniferous iron ores can be reduced without difficulty*.

When the titanium content of the ore is above 5%, not only does it become difficult to reduce the ore at the temperature of the blast furnace, but two further disadvantages occur: firstly, the titanium content of the slag tends to raise its melting point and viscosity; secondly, owing to the large volumes of air passing through the blast furnace, titanium nitrides, which are hard and almost infusible, are formed. At the higher temperature of the electric furnace, and owing to the close control that may be exercised over the process, these difficulties do not arise.

As stated previously, there are vast reserves of titaniferous iron ores in various parts of the world. Most of these contain a higher percentage of vanadium than any other iron ore, and thus the recovery of vanadium from such ores in the electric blast furnace appears to be an obvious undertaking. In electric smelting, the vanadium goes into the pig iron, just as in the blast furnace, and this is usually Bessemerised in acid converters to produce a high vanadium slag.

In Norway, such slags have been successfully produced for several years by the electric smelting of titaniferous Norwegian iron ores. At present, the Union of South Africa is seriously considering the development of its titaniferous iron ores in the Transvaal by electric smelting[6]. The analysis of this ore is shown in Table III. The recovery of vanadium in this case is the foundation upon which the economics of the whole proposal are based. In preliminary experiments, acid converter slags containing 23% of vanadium pentoxide (13% V) have been produced from this ore in a single smelting and blowing operation. According to the proposal, the production of 40,000 tons of pig iron is envisaged annually. Taking 70% as an average yield for vanadium recovery from pig iron (through steel slags resulting finally in vanadium pentoxide), this corresponds to a yearly output of about 400 tons of vanadium, and this would mean that South Africa would become an important exporter of vanadium.

* The Krupp-Renn process, specially designed for low-grade high-titanium iron ores, proved uneconomic because of frequent trouble with the furnace lining; the process was eventually used in Germany for the reduction of nickel ores. There is no information about similar plants that are supposed to be operating at present in Czechoslovakia and the Soviet Union.

Titano-magnetite sands in New Zealand yield a magnetic concentrate containing 0·2–0·3% of vanadium with a V/Fe ratio of 0·3–0·5%. The indicated and inferred resources of this titano-magnetite are 233,000,000 tons, extending along the south coast line of Taranaki. Although there is potential hydro-electric power in the area, no development of this source is envisaged in the near future.[7, 8]

(c) *Production of Vanadium Slags.*

From the pig iron, owing to its high heat of oxidation, vanadium passes almost entirely into the slag in both the Bessemer and basic open hearth processes of steel-making. At this stage, the most important steps have to be taken in order to recover the vanadium economically.

The actual sequence by which oxidation takes place is as follows: firstly, silicon is oxidised, followed closely by vanadium, and thirdly, phosphorus. If, therefore, one could remove successively the slag formed in a Bessemer converter, the slag formed in the early stages of blowing would contain practically the whole of the silicon and a large proportion of the vanadium. At Corby, it has been found that after 9 minutes of blowing, 90% of the vanadium is removed from the metal, and that practically no vanadium remains in the final steel[5]. The process of oxidation of phosphorus can be influenced by the type of lining of the converter and the amount of lime added. Thus, if the slag is deficient in lime to combine with phosphorus pentoxide, there is a reverse tendency for the phosphorus to be reduced back into the metal; this is what actually happens in acid converters.

In an ordinary basic-lined converter, owing to lime additions, the slag volume has a deleterious effect on the vanadium concentration of the product. Because of this, and also owing to the additional dilution of vanadium content by the phosphorus pentoxide, the best results are obtained if the blowing operation is carried out in a converter lined with silica or neutral

TABLE IV.—IRON AND VANADIUM CONTENTS OF SOME TYPICAL BRITISH AND IMPORTED IRON ORES.

	Fe %	V %	V/Fe %
Grangesberg (Sweden)	57·88	0·160	0·28
Kiruna (Sweden)	64·2	0·11	0·17
Gellivare (Sweden)	66·2	0·11	0·17
Wabana (Newfoundland)	50·61	0·05	0·10
Oued Zem (French Morocco)	46·18	0·044	0·10
Chazé Henri, Anjou (France)	52·12	0·031	0·06
Corby, Northants	34	0·055	0·16
Irthlingborough, Northants (Sinter)..	43·04	0·078	0·18
Leicestershire, Eaton Limey	27·0	0·05	0·19
Leicestershire, Holwell	28·7	0·06	0·21
Frodingham, Lincs. (Crosby)	24·66	0·051	0·21

TABLE V.—ANALYSES OF SOME VANADIUM SLAGS.

Source	V %	Cr %	SiO_2 %	CaO %	MgO %	P %	Mn %	Fe %	TiO_2 %	Al_2O_3%	Process	V_2O_5 %
Watenstedt	15·28	2·50	11·74	2·94	1·22	2·46	11·47	30·05	1·33	0·84	Acid	27
Krupp	9·28	5·26	9·32	0·40	1·52	5·73	15·21	29·66			Acid	16·6
Schiffingen	9·65	2·40	16·22	7·55	4·47	2·02	21·76	15·72			Basic	17·3
Mannesmann	7·85	3·42	16·98	4·44	0·80	1·76	27·56	16·18	1·00	0·10	Acid	14
Spigerverk (Norway)	9·30	0·68	30·74	0·60	2·49	1·22	2·04	26·93	3·92	2·09	Acid	16·6

refractories. Optimum results are obtained if the blowing operation is interrupted when most of the vanadium is oxidised and the slag is removed at this stage.

Treatment of Vanadium-Containing Steelworks Slags

Slags best suited for recovery of vanadium are those which have a high silica content and are low in oxides of calcium, magnesium and aluminium. The normal chemical treatment of the slags consists briefly in roasting with soda ash and then extracting with water to remove the soluble vanadate. The extract is then boiled with hydrochloric or sulphuric acid to decompose the vanadate. Sodium chlorate may be added at this stage. The precipitated vanadium pentoxide is filtered and dried. Under present conditions steel slags should contain at least 11% of vanadium pentoxide (6% V), for economical recovery of vanadium by the chemical fusion process. Table V shows the analyses of some typical German vanadium-slags, together with that of the Norwegian slag resulting from the electrically smelted titaniferous iron ores.

Difficulties have been encountered in Germany in the chemical recovery of vanadium oxide from slags; these were due to the phenomenon of "reversibility" or "recovery"[9]. In effect, this means the formation of water-insoluble vanadates, and it happens especially when the roasting mixture is allowed to cool slowly. It is probably due to the formation of an insoluble oxide of tetravalent vanadium, which has been actually detected by X-ray. A great deal of research has been carried out in Germany on this problem, and some promising results have been achieved. In general, however, the normal practice has been to return the insoluble residue to the blast furnace to recover the remaining vanadium.

Possible Methods of Vanadium Recovery from British and Imported Iron Ores

A pre-requisite of all recovery processes is the availability of suitable vanadium-containing iron ores. Such ores have been described in the earlier sections of this paper; Tables III and IV show analyses of some of them. In some cases the iron ore has been considered to contain sufficient vanadium to warrant its recovery when its V/Fe percentage was higher than 0·1. During the war, in Germany, for example, it was necessary to recover vanadium from certain Minette ores, some of which contain even less vanadium. In the United Kingdom, however, with due regard to present conditions, the special difficulties which an extensive recovery would entail, and its possible effect on steel output, it is thought that steelworks which exclusively process ores with a V/Fe percentage below 0·16% need not consider vanadium recovery.

Those who mix ores from various origins, on the other hand, may well be asked to see that high-vanadium ores are kept separate and put through the blast furnace with a view to making a special vanadium-containing pig iron suitable for further treatment. Generally, the aim should be to produce a pig iron containing at least 0·2% of vanadium.

Production of Vanadium Slags

In Germany, where steelmaking was based mainly on Bessemer converters, it was not so difficult to introduce certain steps in steelmaking in order to ensure a satisfactory vanadium recovery. This was done mainly by recovering the slag formed in the early stages of blowing. However, even in Germany, the interruption of the blowing operation was not favoured, as it seriously interfered with steel production. In the United Kingdom on the other hand, there are only two important plants using the Bessemer process (at Workington low-phosphorus hematites are treated and, although they employ the acid Bessemer process, the ores contain little or no vanadium), and the main volume of steel production, is carried out by the basic open-hearth method.

At first sight it would appear, therefore, that in Britain the recovery of vanadium from iron ores presents an almost insuperable problem. However, this is fortunately not the case, for the following reasons:—

(a) owing to the enormous quantities of vanadium-containing ores that are being processed in the various steelworks, even a limited recovery programme would be adequate to meet requirements;

(b) local ores where the Bessemer process is in use are comparatively high in vanadium; and

(c) certain basic open hearth steelworks would benefit from a preliminary blowing of their pig iron in order to de-siliconise it. If adopted, this process could be combined with the recovery of vanadium.

In the following, the suggested recovery processes are dealt with according to whether steel is made by the Bessemer process, or in the basic open-hearth furnace.

(a) Recovery by the Bessemer Process.

Probably the most efficient recovery would be by duplex blowing; i.e., a preliminary blowing in an acid-lined converter, followed by blowing in the normal basic-lined converter. This, however, would seriously interfere with steel output; the interruption of the blowing operation to remove the vanadium-rich slag would be objected to for the same reason. In Germany, it was found that the converter spittings and roof sediments were particularly high in vanadium content; for example, basic Bessemer spittings from a Minette ore contain between 0·5 and 1·5% of vanadium[10]. In Britain, the ores used at Bessemer plants are superior, from the standpoint of vanadium content, to the Minette ores, and it is reasonable to assume that these sediments and spittings would have a far higher vanadium content. It is not expected, however, that there would be sufficient quantity of such material and it would, therefore, have to be mixed with ordinary basic slag, which at Corby contains 1–1·2% of vanadium pentoxide (0·6–0·7% V)[5]. If, in addition, every fourth or fifth converter charge were blown for 3 to 5 minutes

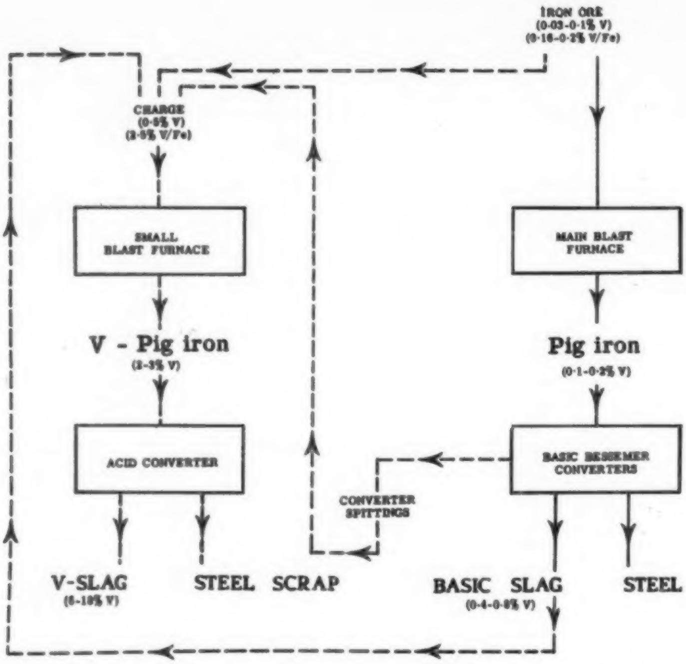

Fig. 1.—Possible method of vanadium recovery in Bessemer plants.

only, and the slag were collected, that would yield additional quantities of high-vanadium slags without seriously interfering with steel output. A special charge could be made up by adding a small quantity of high vanadium ore to this material. This charge with a high V/Fe ratio could be reduced in a small blast furnace specially built for the purpose. A blast furnace with a capacity for treating 70 tons of slag per day would be adequate for this purpose. Alternatively, one blast furnace may be set aside for the purpose of making vanadium pig iron.

The vanadium-rich pig iron would subsequently be blown in acid-lined converters until the end of the vanadium blow-out; the slag thus yielded is expected to contain between 10 and 30% of vanadium pentoxide (6 and 18% V), according to the vanadium content of the vanadium pig iron. When the vanadium slag has been removed, blowing might be continued, and the converters would produce a high-grade steel scrap. (See Flow-Sheet, Fig. 1.)

(*b*) *Recovery of Vanadium in Basic Open Hearth Steelworks.*

The basic open hearth process of steelmaking creates added difficulties regarding vanadium recovery. There is less control over the process, so far as vanadium recovery is concerned, than with Bessemer converters, and the slag formed will normally contain a lower proportion of vanadium. Ordinary basic open hearth slags produced from vanadium-bearing ores contain approximately 0·5–1·2% of vanadium pentoxide (0·3–0·7% V).

If slags of this type were put back into the blast furnace, a comparatively high vanadium pig iron could be made, but it would not be possible to get the best results of final recovery, if such pig iron were to be processed again in the basic open-hearth furnace. There are, however, two possibilities of recovering vanadium before the pig iron enters the open-hearth furnace, namely by (1) preliminary blowing in an acid converter and (2) allowing the pig iron to run through a deep trough or tunnel lined with silica bricks, and blowing jets of air over it.

Owing to difficulties experienced in certain basic open hearth steelworks with the formation of highly viscous siliceous slag, it has been suggested that the pig iron should be de-siliconised before it enters the basic open-hearth furnace. Silicon, with its high heat of oxidation, is oxidised rapidly, vanadium following it closely. A preliminary blowing, therefore, would remove practically all the silicon and the greater part of the vanadium; much the same result could be achieved by adding hammer scale and passing pig iron, under oxidising conditions, through a trough, though, in this latter case, more vanadium is likely to remain in the pig iron. (See Flow-Sheet, Fig. 2.)

(1) *Preliminary Blowing in an Acid Converter.*—The suggestion is to apply a preliminary blowing of three to five minutes in a silica-lined converter, discharge the concentrated vanadium-siliceous slag and transfer the pre-blown pig iron into the basic open-hearth furnace. The resulting slag may have to be put back once more through a special blast furnace, with some high vanadium ore, and smelted into a special vanadium pig iron. When this is blown in an acid converter for a short time, it is expected to yield a vanadium slag containing between 16 and 29% of vanadium pentoxide (9% and 16% V), according to the ores used.

(2) *Trough Process.*—This process was developed in Germany at Volklingen[10]. The vanadium pig iron is passed through a cylindrical steel tube of about 5 feet external diameter lined with refractory material. As the metal flows in a thin stream through this tunnel or trough, it is subjected to the action of air jets over the surface of the metal; hammer scale may also be added. The operating speed of such a trough is given as about 20 tons of pig iron per hour. The disadvantage of this process is that it is difficult to control the temperature of the pig iron, and there may be trouble due to rapid cooling. On the other hand, it does not require a great deal of extra cost for special plant.

In one modification of the process in Germany, the trough was lined with hammer scale, with or without iron ore in powder form. The process is less efficient from the standpoint of vanadium recovery, probably because there is insufficient time for the air, which is blown over the surface of the molten metal, to diffuse through and oxidise all the vanadium. This process may nevertheless be worth considering because of its low cost.

Recovery of Vanadium from Ordinary Basic Slag

In Germany towards the end of the war, the suggestion was made to reduce *low vanadium* Thomas slag in the electric furnace with coke[11]. Thereby, steelworks would

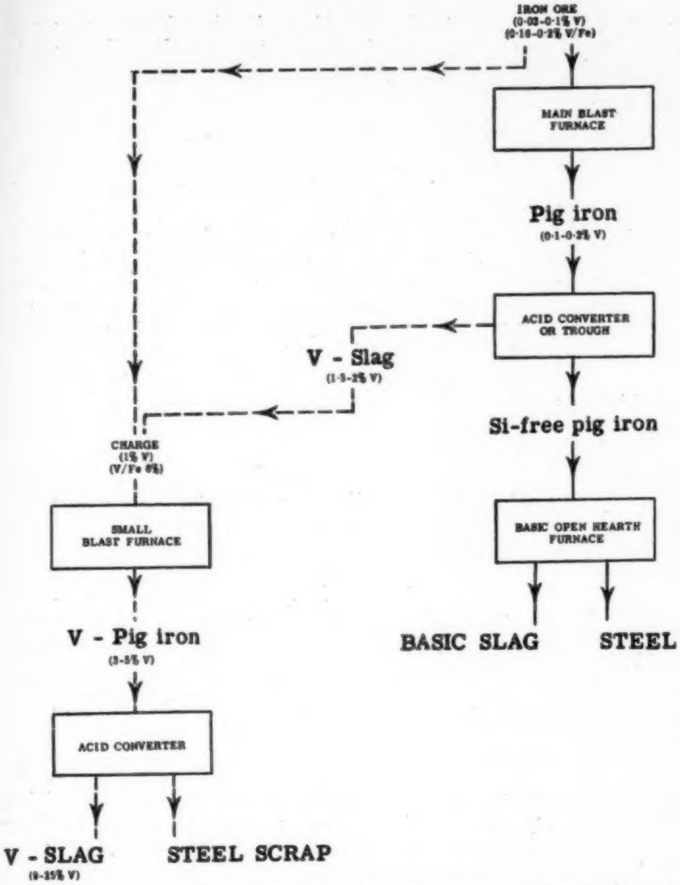

Fig. 2.—Possible method of vanadium recovery in open-hearth plants.

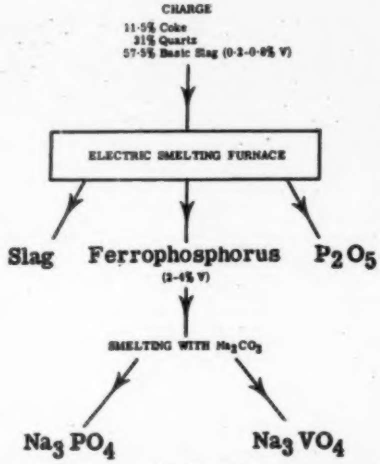

Fig. 3.—Method of vanadium recovery from ordinary basic slag in the electric furnace.

be relieved of the task of vanadium recovery ; that is, it would not be necessary to produce a special vanadium pig iron, or to take special steps during the blowing operation. In Table VI the compositions of some typical basic slags together with a German Thomas slag are given. The compositions of the high-phosphorus British slag and of the Thomas slag are similar.

If such slags are treated in the electric furnace, about half of the phosphorus is obtained as elementary phosphorus, which is oxidised to phosphorus pentoxide ; the rest combines with the iron to form ferro-phosphorus. The latter contains the vanadium, which undergoes a five-fold concentration on going from the slag into the ferro-phosphorus. The following analysis shows the composition of the ferro-phosphorus regulus obtained in an experimental 13 kW. electric furnace from a Thomas slag, the composition of which is shown in the last column of Table VI. According to the proposal, this ferro-phosphorus was to be fused with soda ash, and both the vanadate and the trisodium phosphate were recovered. (See Flow-Sheet, Fig. 3.)

The percentage composition of ferro-phosphorus resulting from the treatment of Thomas slag in the electric furnace is :—

Fe	P	Si	Mn	V
71·55	22·61	0·85	3·04	2·37

If it were possible to make a selection of certain basic slags containing at least 0·8% of vanadium (e.g., as from Corby), then, according to this process, the resulting ferro-phosphorus may contain about 4% of vanadium. Moreover, the production of two valuable chemicals—i.e., phosphoric acid and trisodium phosphate—simultaneously with vanadium oxide, makes this process worth considering, especially where cheap electric power is available. A further advantage is the supposedly higher vanadium recovery from ferro-phosphorus, than from high-vanadium slags. The disadvantages are : the necessity of treating large quantities of slag in the electric furnace and the comparatively low vanadium content of the ferro-phosphorus, which must undergo the usual chemical processing. Cost calculations, based on actual experiments, show that, even assuming peace-time conditions, this was considered to be an economic proposition—at least so far as Germany was concerned.

Conclusions

Vanadium mineral resources of the sterling area are wholly inadequate to meet the needs of this country and Western Europe. Supplies of vanadium-containing oil refinery residues, hitherto an important source, appear to be less readily available. The U.K. is, therefore, almost entirely dependent on vanadium minerals from South West Africa.

Vanadium is not being considered by the International Materials Conference, and the United States herself imports considerable quantities from Peru.

TABLE VI—ANALYSES OF TYPICAL BASIC SLAGS.

	(a) British Basic Slags				(b) German Thomas Slag
	%	%	%	%	%
Phosphoric Acid (P_2O_5) ..	8·12	10·29	14·18	16·56	17·72
=Total Phosphates	17·74	22·45	30·96	36·15	
Lime, as CaO	46·22	46·66	45·70	45·50	49·25
Iron Oxide	11·89	12·00	10·80	11·10	13·85
Manganese Oxide	3·96	5·51	5·44	4·90	2·55
Magnesia	6·71	6·63	6·20	6·15	3·48
Sulphur	0·20	0·23	0·27	0·17	·?
Silica	16·20	13·50	11·60	10·35	8·38
Alumina, etc.	4·25	5·18	5·81	5·27	1·03
Vanadium Oxide (in terms of metal)	Between 0·2 and 0·8				0·47

Source : (a) British Basic Slag Producers' Association ; (b) German Reports.

Most British and some imported iron ores contain a small percentage of vanadium, which tends to be concentrated in the steel slag during the process of iron and steel production. Certain steps have been mentioned in this paper which, if adopted, would preserve this vanadium and increase its concentration in the steel slag sufficiently to make its subsequent extraction an economic proposition.

The steps suggested may be summarised as follows:

(1) The segregation of all iron ores with a high vanadium content, to make a suitable vanadium-containing pig iron in the blast furnace, with about $0 \cdot 2\%$ of vanadium. This may be achieved by using charges of ore with an average V/Fe ratio greater than $0 \cdot 16\%$.

(2) In the *Bessemer Process*:

- (*a*) To collect converter spittings and similar high-vanadium slags and, by mixing these with some ordinary Bessemer slag and iron ore, to make up a charge for reduction in a small blast furnace specially built for this purpose; or
- (*b*) To install acid converters and blow the vanadium pig iron obtained in this small blast furnace, and thus make a high-vanadium steel slag.

(3) In the *Basic Open-Hearth Process*, to install a duplex process; i.e., to remove the silicon and the vanadium from the pig iron by oxidation before it enters the basic open hearth furnace, either by—

- (*a*) blowing for a short time in acid converters; or
- (*b*) running the molten pig iron through a trough, adding hammer scale and blowing jets of air over its surface.

Owing to the vast quantities of vanadium-containing ores handled by the steel industry, even a limited recovery programme would be adequate to meet the immediate shortage. The cost of production of such high-vanadium steelworks slags can, of course, hardly compete with naturally occurring high-grade vanadium ores. The present shortage of vanadium, however, probably justifies drastic steps being taken, and the best solution lies in vanadium recovery from iron ores.

This work was carried out as part of the programme of the Intelligence Division of the Department of Scientific and Industrial Research and is published by permission of the Head of the Division, Dr. Alexander King.

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Further Modernisation of the Tinplate Industry

In the course of a speech at a dinner given to the Mayor, Aldermen and Councillors of Port Talbot, Glamorgan, on Saturday, 29th November, 1952, Mr. E. H. Lever, Chairman of The Steel Company of Wales, Ltd., and Richard Thomas and Baldwins, Ltd., said that the Iron and Steel Corporation had now advised him that they formally approved the Steel Company of Wales' programme for the further modernisation of the tinplate industry.

The principal feature will be a new five-stand cold reduction mill, similar to the one at Trostre, to be erected at Velindre, Llangyfelach, between Swansea and Llanelly. Other developments included in the programme are additional coke ovens, an extension of the sintering plant, a fourth blast furnace, four open hearth steel melting furnaces and sundry rolling mill equipment and ancillary plant to be installed at Margam, Port Talbot, and Abbey Works. At Trostre, additional annealing, temper-pass and tinning plant will be installed. The whole programme is estimated to cost about £40,000,000 on present prices. It is estimated that the new Velindre Works will produce in the region of eight million basis boxes a year.

Mr. Lever said that the decision was timely because world conditions were rapidly changing and it was, therefore, of paramount importance that the total modernisation of the industry should be pressed on with all speed.

Until this vast new extension is brought into operation a large volume of output from the old type plants would still be required. The problem of redundancy of labour which may arise from the ultimate closing down of these old type plants will as heretofore be a matter of close consultation between the Company and the Trades' Unions concerned.

Corrosion-Resistant Materials in Marine Engineering

Corrosion problems in marine engineering vary considerably, but it can be said that materials are available to resist practically all conditions of service. Although the use of such a material may not always be justifiable, by slight re-design and by choosing a more resistant alloy, economies and improved life are often possible. A booklet dealing with this subject, based on a paper presented to the Institute of Marine Engineers by L. W. Johnson and E. J. Bradbury, has recently been published by The Mond Nickel Co., Ltd.

After a brief introduction to the theoretical aspects of corrosion and a consideration of causes—products of combustion of fuel, boiler water impurities, steam, sea water, the atmosphere—much useful data is given on the corrosion-resisting materials commonly in use. The materials covered include copper and special brass and bronze alloys, cupro-nickel, aluminium alloys, corrosion-resisting and heat-resisting steels and cast irons, and protective metallic coatings. A useful table shows the relative abilities of a wide range of copper alloys to resist different forms of attack by sea water. A comprehensive bibliography is included. Copies of this publication may be obtained free from The Mond Nickel Co., Ltd., Sunderland House, Curzon Street, London, W.1.

Push-Button Controlled Wheel Forging and Rolling Plant

Close up of the wheel rolling mill in action.

AN outstanding example of the application of mechanical handling in heavy industry is provided by the new wheel forging and rolling plant which recently went into production at the Trafford Park works of Taylor Bros. and Co. Ltd., the largest producers of railway wheels in the British Empire. The new plant has reduced labour requirements by about a half, and has increased output per man-year to almost three times as much as in the old plant. Furthermore, the work is much less arduous and dirty, the whole process being controlled by eleven operators sitting at switchboards in air-conditioned cabins. Each operator has at his disposal the equivalent of 480 horse-power.

This plant, which produces wheels ranging from 24 in. to 50 in. diameter on tread at the rate of 60 an hour, and which cost over £1,000,000, has been financed entirely by English Steel Corporation Ltd., Taylor Bros. and Co. Ltd. being one of the members of the English Steel Corporation Group of Companies. Following on the installation of new plants in Sheffield for the improved production by E.S.C. of railway springs and cast steel bogies and automatic couplers, the new wheel plant is further evidence of the importance attached by the E.S.C. Group to the production of railway materials, of which more than 50% is exported to countries all over the world.

In conjunction with the Consulting Engineer, Mr. E. Homer Kendall, the layout of the plant and the design of the equipment was completed by the Engineering Department of Taylor Bros. and Co. Ltd., under the personal control of Mr. J. H. Ellis, Assistant Chief Engineer in charge of Development. The major items of plant comprise a 69-ft. diameter rotary hearth furnace, an 8,500-ton forging press, a 1,000-ton punching press, an electrically driven rolling mill, a 2,000-ton dishing press and ancillary fully automatic handling plant specially designed to ensure fast production, together with an appreciable decrease in labour as compared with existing wheel plants. Apart from the furnace and electrical gear, most of the plant has been designed and built within the E.S.C. Group, the Elswick works of Vickers-Armstrong Ltd. playing a major role. The electrical side, including the operators' control panels, was the responsibility of Metropolitan-Vickers Electrical Co. Ltd.

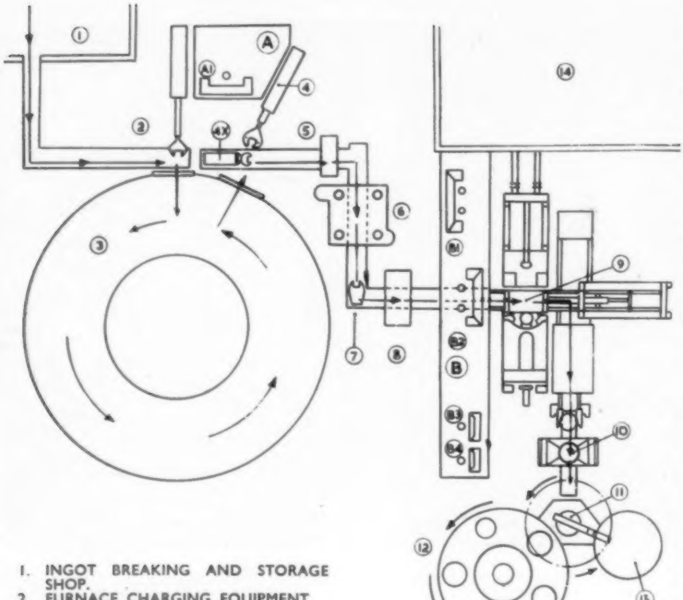
Separate 6,600 volt incoming supply cables and switchgear have been installed and the plant has self-contained hydraulic, cooling water and compressed air services. These, together with the mill motors, generators, oil-hydraulic and lubricating equipment, are housed in a separate substantial building of modern design. With the object of reducing production delays and maintenance costs to a minimum, particular attention has been paid to the installation of single purpose equipment designed to give reliable service under conditions of continuous operation.

A distinctive feature of the plant is the extensive use of special manipulating machinery fitted with individual oil-hydraulic units. Economies in power have been effected by the use of air-loaded accumulators in conjunction with the main hydraulic plant to regulate the operating pressure in accordance with the section being made. Power-operated controls and automatic equipment are incorporated with the object of eliminating physical fatigue and making possible the continuous maintenance of high rates of production.

Charging the Furnace

Octagonal or duo-decagonal cross-sectioned ingots, of width $12\frac{5}{16}$–18 in. across the flats, are divided into correctly sized blocks by a Scrivens ingot breaker, after first flame nicking, or, in the larger sizes, partially parting in a lathe. The blocks pass to stock down a roller conveyor incorporating a weighing section, a further roller conveyor being used to transport blocks from stock to the furnace charging machine. The latter consists of a cable-driven carriage mounted on a fixed but adjustable bridge, the peel hoisting mechanism and the oil-hydraulic equipment for the gripping motion being mounted on the carriage.

The charger is arranged automatically to complete a cycle of operations initiated by the operator after he has



1. INGOT BREAKING AND STORAGE SHOP.
2. FURNACE CHARGING EQUIPMENT.
3. ROTARY HEARTH FURNACE.
4. FURNACE DISCHARGING EQUIPMENT.
4x BLOCK TRANSFER BOGIE.
5. HYDRAULIC DESCALING STAND.
6. 8,500-TON FORGING PRESS.
7. PRESS UNLOADER.
8. 1,000-TON PUNCH PRESS.
9. WHEEL ROLLING MILL.
10. 2,000-TON DISHING PRESS.
11. HYDRAULIC JIB CRANE.
12. RIM CHILLING MACHINE.
13. FINAL LAYDOWN STATION.
14. MOTOR AND PUMP ROOM.

A. FURNACE CONTROL ROOM.
A1 HANDLING OPERATORS DESK.
B. MAIN CONTROL ROOM.
B1. PRESS CONTROL DESK.
B2. MILL CONTROL DESK.
B3. PRESS CONTROL DESK.
B4. CHILLER CONTROL DESK.

Layout of the plant.

indexed the rotary hearth of the furnace. With the tong head resting on a centring cam and the block lying on the conveyor against a stop, the cycle commences with the lifting of the furnace charging door. The block is gripped at a fixed distance from its base, and as the peel structure is raised the tongs rotate to bring the block axis into the vertical position and the base of the block to a fixed distance above hearth level. The carriage then moves to the extreme forward position (in order to place the first block in the innermost position on the furnace hearth), lowers the block, releases it and returns to its original position with the tong head resting on the centring cam. The cycle is then repeated and a second block is charged, the forward travel of the carriage being sequence controlled so as to enable a radial row of blocks to be charged, the number being variable up to a maximum of seven. After the seventh or outside bloom has been charged, the furnace door is closed and peel and tong-head cooling sprays are brought into action.

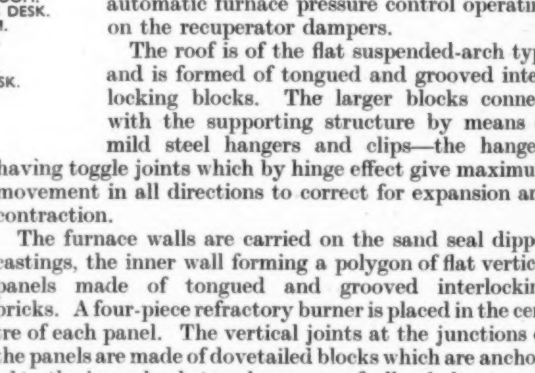
The Furnace

Built by the Salem Engineering Co. Ltd., the 69-ft. diameter furnace is the largest of its kind in the country and has a rated heating capacity of 40 tons of steel an hour at the maximum temperature of 1,280° C. The charging and discharging doors are set at an angle of 30° and there are two suspended baffle walls built radially across the furnace between the doors. Small doors in the inner and outer walls, between the baffle walls, facilitate cleaning of the hearth whilst the furnace is in operation. The furnace has four firing zones and is fitted with 39 steam atomising burners designed for heavy fuel oil burning. Preheated air, obtained by passing the waste gases through two Newton Chambers needle-type metallic recuperators, is used in the second and third zones, and atmospheric cold air in the pre-heating and soaking zones. The hearth, which is of the Salem floating type, is driven at diametrically opposed points by a 27 h.p. motor coupled through a differential gear, line shafting and reduction gears. The hearth is carried on 108 wheels fitted with taper roller bearings running on circular rails.

Fifty-three rows of blocks are arranged in the 330° between the charging and discharging doors, giving a maximum of 371 blocks in the furnace at any one time. The rows of blocks are carried through the heating and soaking zones of the furnace by progressive indexing of the hearth at a rate corresponding to the output of the plant. At a production rate of 60 pieces per hour, the heating time of the blocks is approximately $6\frac{1}{4}$ hours. The furnace is equipped with Honeywell-Brown "Electronik" temperature controllers and Electroflo fuel/air ratio controls on each zone, and with Electroflo automatic furnace pressure control operating on the recuperator dampers.

The roof is of the flat suspended-arch type and is formed of tongued and grooved interlocking blocks. The larger blocks connect with the supporting structure by means of mild steel hangers and clips—the hangers having toggle joints which by hinge effect give maximum movement in all directions to correct for expansion and contraction.

The furnace walls are carried on the sand seal dipper castings, the inner wall forming a polygon of flat vertical panels made of tongued and grooved interlocking bricks. A four-piece refractory burner is placed in the centre of each panel. The vertical joints at the junctions of the panels are made of dovetailed blocks which are anchored to the inner buckstays by means of alloy bolts, to pre-



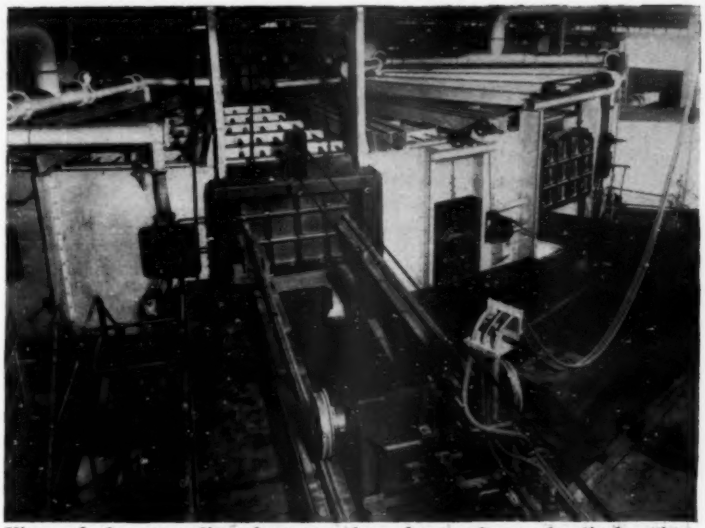
Interior view of the furnace showing the charging door, burner ports, gas outlet and one of the curtain walls between the charging and discharging doors.

vent outward movement due to expansion.

The furnace foundation has been designed as an open pit about 7 ft. deep to ensure adequate ventilation under the hearth and to prevent overheating of the steel structure and wheelbearings: lubricating of the latter is also facilitated.

Furnace Discharging Machine

The heated blocks are removed from the furnace by a high bridge type discharging machine of 1 ton capacity. The machine is of a similar basic design to the charging machine—having a cable driven carriage in which is mounted a rocking peel structure. Travel of the carriage to the predetermined billet position is controlled by a multiple cam type limit switch with vernier setting adjustment and "slow down" and "stop" rings for each billet position. To commence the discharge of a row of blocks, the carriage travels to the outermost hearth position with the peel lowered. Completion of travel initiates gripping, which is followed by a hoisting of the peel, withdrawal to an air-operated elevator ram, and a lowering of the peel. If the elevator is in the raised position, the release of the grips and subsequent lowering of the elevator take place automatically together with a closing of the furnace door. At the lower level the block is gripped by the transfer tongs and the operator then initiates its transfer to the descaler and press. The discharging machine may then be restarted; after the furnace door has been rehoisted, it commences its inward travel again and completes its cycle by withdrawing and holding a second block over the elevator until the latter is automatically raised following the return of the transfer car to the furnace. The cycles are repeated until the innermost block in a radial row has been discharged. A system of signal lamps on the control desk shows the operator at a glance how many blocks remain to be charged and discharged at any time, and the hearth is not indexed until a clear space is indicated at the discharging position. Complete sequence controls on each machine automatically reset for the next cycle of operations. A rotating dial mounted outside the control room is geared to the hearth and is used for maintaining continuous indications of the progress of the blocks through the furnace, and of the operation of the hearth indexing limit switches.



View of the annealing furnace taken from above the discharging machine. The charging door is on the right with the cleaning door between the charging and discharging doors.

Transfer Car and Descaler

The four-wheeled transfer car runs along a 67 ft. rail track from the elevator near the discharger to the 8,500-ton press, passing through the hydraulic descaler on its way. The block is carried in grips at the front and track switches are provided for slowing down the car prior to it entering the descaler, for operating the descaler control valve, and for finally stopping the car when the block is over the 8,500 ton press slabbing die. Similar switches are used at the furnace end of the track for slowing down and stopping the car on its return journey. Release of the block from the car grips, which is followed by the automatic return of the car to the furnace, is controlled by the press operator.

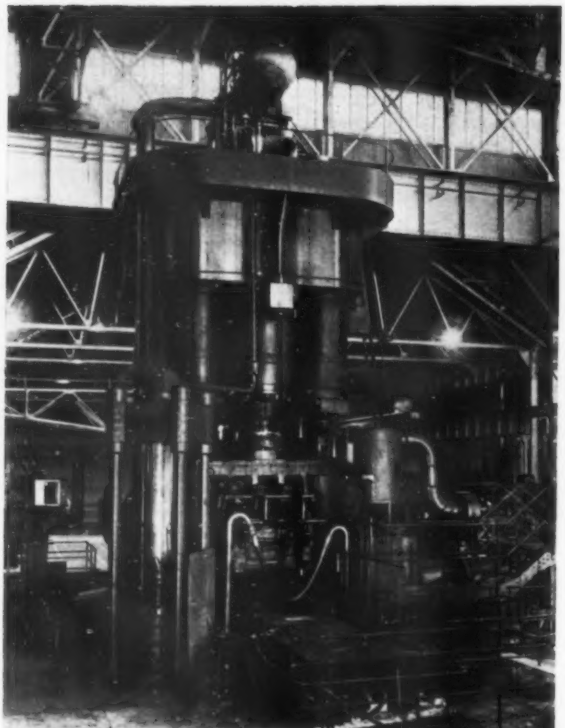
The hydraulic descaling machine has six Harland-Aldrich No. 2 nozzles each of 27·2 gals./min. capacity mounted in an adjustable header pipe above the block, and six similar nozzles in a fixed header pipe arranged 6 in. below the lower surface of the block. A Schneible Multiwash Collector with extractor fan is fitted to prevent the steam generated from obscuring the view of the press operator. Canal water at a pressure of 1,500 lb./sq. in. is used for descaling.

8,500-ton Press

The block is slabbed down on the forging press between flat dies until the required thickness is obtained, gripped in the centring arms and transferred to the lower forging die. The upper sliding table is then moved to bring the top forging die under the centre line of the press. The second forging operation is completed under intensified pressure, and during this process the next block to be forged is placed on the slabbing die. The lower sliding table is moved back to bring the completed forging over the stripping ram and the following block under the press crosshead.

The press is fitted with four cylinders having a diameter of $33\frac{3}{8}$ in. and a stroke of 36 in., the daylight between the sliding tables being 67 in. Top and bottom tables have travels of 5 ft. and 9 ft. 6 in. respectively. The columns are at 8 ft. centres and have a minimum diameter of 26 in. Fabricated structures made from castings and plates serve as press bed and press crown, whilst the crosshead is a one-piece steel casting. The crosshead bushes, which are lined with white metal, and all main glands are fed with grease from a Trabon automatic system.

The hydraulic intensifier, which has a ratio of 2·87 : 1 gives a pressing stroke of 6 in. At 5,600 lb./sq. in. intensified pressure, the maximum load on the press is 8,500 tons; this load can be reduced for smaller forgings by reducing the pressure in the air-loaded accumulators. The press is equipped with a large prefill tank and surge tank, the latter being mounted directly over the prefill valves on the press crown to reduce cavitation in the cylinders when lowering the crosshead under prefilling conditions. All the main hydraulic valves are arranged in the press foundation between the press and the



The 8,500-ton wheel forging press.

control room, and a pressure switch is installed to prevent operation of the intensifier before full accumulator pressure has been developed in the main cylinder.

The control gear is electric-hydraulic, using V.S.G. auto-controlled pump and solenoid-operated pilot valves to direct oil to servo cylinders mounted under the main valve spindles. With this system, it is possible to group all the press controls on a desk so that this stage of forging is under the control of one operator who is protected from heat, steam and noise and seated in the main control room.

On being lifted from the bottom die by the stripping ram, the forging is gripped by a special machine, lifted further and slewed through 90° before lowering on to a 4-section live roller table which runs through the punching press and on to the rolling mill.

The forging, on arrival at the upstroking type punching press, which has a rated capacity of 1,000 tons and a punching stroke of 9½ in. at a speed of 3 in./sec., is centred by an adjustable stop mechanism, and lifted on the live rollers by the lower boss clamping die. Continued upward movement of the crosshead first clamps the boss and presses the hub against the rigid punch. When the punch has completely penetrated the boss, the crosshead is stopped by limit switch control and the forging is lowered on to the live rollers and proceeds to the mill. The control gear is similar to that for the 8,500 ton press.

The mill loading mechanism is mounted under the main control room, its main supporting frame being pivoted at the mill end and suspended at the other end from a curved track. This arrangement facilitates the alignment of the machine in relation to the mill rolls. A simple carriage, activated by a hydraulic cylinder carries a self-centring grip mechanism which is rotated to enable the grips to clear the wheel forging. The travel stroke is arranged to take the bloom from the end of the roller table to the centre line of the mill, where the forging is deposited on forks attached to the roll carriages.

The Wheel Mill

The forged rim section is reduced in area in the mill and formed to the required shape by means of driven edging rolls which bear on the front and back faces and on the inside contours of the rim, and by the load on two pressure rolls which bear on the tread and form the flange. The mill, which is of Taylor–Kendall design, has also a driven main roll, mounted in an adjustable carriage, and two guide rolls mounted on a separate carriage but sliding on the same ways as the main carriage. The hydraulic pistons for the guide roll carriages are attached to the main roll carriage to give movements relative to that carriage. The mounting arrangement of the guide rolls permits their retraction behind the main roll to facilitate loading and unloading of the mill. The arrangement adopted reduces the handling time at the mill and simplifies the handling equipment. Pressure regulation on the carriages during rolling controls the formation of the section and the rate of expansion of the wheel diameter. Indicating equipment is built into the mill to show the inside diameter of the rim, and mill setting scales are mounted on the side of the main stand for rapid setting-up to suit varying rim widths and roll diameters.

Each carriage has a self-contained oil-hydraulic unit comprising large volume low pressure pumps for rapid traverse duties and small volume high pressure pumps for maintaining pressure during rolling. The pumps, together with the electrically operated control valves, are mounted on tanks which contain all the system connections. Only the cylinder piping and remote control drains are located outside the motor room. Cylinder pressures are remotely controlled at the mill desk by the adjustment of venting valves coupled to Vickers–Detroit hydrocushion relief valves which are mounted on the operating side of the cylinders.

The top and bottom edging rolls are each driven by a 400 h.p. D.C. motor having a speed range of 690 to 860 r.p.m. These motors during rolling are coupled in series across a 480 volt generator. The motors drive through a primary reduction gear of 38/136 ratio and wobbler shafts which are coupled horizontally to 20T. 1·4184 D.P. spiral bevel pinions engaging 27T. wheels mounted on the roll shafts. The edging roll shafts are inclined at 35° to the horizontal plane and are mounted in quill castings which also form the bevel gear casings. At the roll end of the shafts are 4-row Timken "balanced proportion" type roll neck bearings. Steep angle taper roller bearings are mounted in a cartridge behind the bevel gears. The input bevels are mounted in the quills on stub shafts carried in cartridge-mounted taper roller bearings. The main roll, which is the only other driven roll, is carried on a tapered extension of a vertical shaft mounted on a Timken 2-row "balanced proportion" roll neck bearing with a double row taper roller bearing at its lower end. A single helical gear wheel is mounted on the roll shaft and engages a pinion on a vertical intermediate shaft which also carries a spiral bevel wheel. A bevel pinion is mounted horizontally on a

shaft coupled to a 150 h.p. mill motor having a speed range of 460 to 1,150 r.p.m. This motor is fed from a separate generator mounted on the main set and has a "shovel" characteristic to prevent it taking load from the edging roll drive motors and overloading itself. All driven roll speeds are independently variable and the overall mill speed is also adjustable to maintain pre-set roll speed ratios modified to suit the various roll diameters.

The primary gearbox, edging roll quills and main roll gearbox are fed with lubricating oil from a De Laval circulating unit made by the Denco Engineering Services, Ltd., and installed in the motor room basement. The unit is fitted with duplicate gear-type pumps, a motor driven filter, steam heating coils and oil cooler. It has automatic control equipment to bring the second pump into operation in the event of loss of pressure and is provided with audible warnings for excessive pressure. The mill motor controls are interlocked with the lubrication system to prevent the starting of the mill until the lubrication system is in operation.

General view of the wheel rolling mill. The operators are seated in the control cabin overlooking the mill, and on the far side of the control cabin the press operators can be seen.

The three roll carriages slide on round ways and are fitted with bronze bushes pressure-fed with grease from a Trabon automatic reversing lubricating system. The bushes are fitted with wiper seals. It is claimed that this method of mounting the carriages facilitates effective lubrication and maintenance of roll alignments with consequent improvement in the consistency of product dimensions.

Both edging roll quills are mounted on pivot shafts having an eccentric centre portion for quill adjustment. Rocker bars at the roll end of the quills are connected through adjustable pitman arms to the screwdown crankshafts. The crankshaft for the lower roll is connected through a wormgear to a 5 h.p. mill motor forming a drive for retraction of the roll. The upper quill is similarly connected to a 20 h.p. D.C. mill motor of 450/1,200 r.p.m. This motor is coupled to a Metadyne set having a 35 h.p. driving motor, a 0/17 kW. 0/230 volt generator, a 5 kW. 250 volt exciter, and a 600 watt 150 volt Metadyne exciter. Controls are provided to give "instantaneous" edging, which produces a parallel wheel web, or a "continuous" edging to produce a tapered web by the use of an electronic ratio controller which is coupled to the wheel growth dial. A separate controller is provided for manual adjustment of the top screwdown motor.

Interior view of the control cabin showing the control desks for the mill and presses.

The Dishing Press

The wheel, on completion of the rolling, is transferred to a flat-topped car by an unloading mechanism similar in design to the one used for loading the mill. The car, which is cable driven, takes the rolled wheel over the bottom die of the dishing press. A swinging arm is brought into position by the dishing press operator who then returns the car to the mill end of its track. The car in returning slides from underneath the wheel which is restrained by the stops and the wheel falls on to the die. The lower sliding table of the press is then moved, bringing the die under the crosshead ready for the next forging operation.

The dishing operation changes the web of the wheel from a flat disc to a cone shape and sets the boss offsets. The press has a two-piece top die mounted in a holder on the crosshead and has a bottom sliding table having a stoke of 6 ft. on which are mounted the bottom dishing die and a stripping stool. The press has a rated capacity of 2,000 tons at an accumulator pressure of 2,200 lb./sq. in. intensified to 6,000 lb./sq. in. on a single ram $31\frac{5}{8}$ in. diameter $\times$ 2 ft. 7 in. stroke.

The movement of the wheel after dishing depends on the subsequent treatment. Certain classes of wheel are chilled on the tread before cooling, whilst others are allowed to cool normally; a proportion of the latter are finally hardened and tempered. A 1-ton hydraulic jib crane with automatic tongs fitted to its rigid lifting column, is so located that the dishing press stripping stool, the rim chilling machine loading station, and a final laydown station are at 120° intervals around the 14 ft. diameter circle described by the jib. Prior to leaving the stripping stool, the cast number of the steel is stamped on the wheel.

Rim Chilling Machine

This machine consists of five chilling stations mounted on an electrically-driven turntable 14 ft. in diameter. At each chilling station is placed a set of three tapered rolls, one of which is driven by an electric motor mounted inside the turntable hood. Box-section spray rings are mounted on the roll housings and coupled to a pump located inside the turntable hood through a solenoid operated valve. This pump, rotating with the table structure, draws water from an annular tank located around the vertical post which carries the weight of the whole machine. At the centre of each chilling station is a set of loading forks under which is mounted a live roller centre. At the point of intersection of the spray ring pitch line and the jib crane radius, a fixed cam is arranged to lift the forks above the level of the live rollers in readiness to receive the wheel from the jib crane. After the wheel has been deposited on to the forks the main turntable is indexed one-fifth of a revolution, lowering the forks and leaving the wheel on the rotating rollers. The water spray valve of that station is automatically switched on together with the spray timing device which holds the spray valve open irrespective of further movement of the station. As the first station is carried away from the loading point the fifth station goes into the loading position and the wheel is lifted from the live rolls by forks ready for transference by a hydraulic jib crane to the final laydown position. The water from the spray rings runs down the hood at the main turntable structure into an outer annular tank fitted with overflow pipes which act as a return to the main drainage sumps. The outer casing of the chiller dips into the return water tank forming a water seal and the underside of the hood is ventilated by means of a fan which is mounted on the centre of the main hood structure. This flow of water is used to assist the flushing of scale from the mill and press foundations. The main turntable drive consists of a 10 h.p. motor with brake, coupled by means of an extension shaft to a worm gear unit mounted underneath the turntable hood—a pinion on the wormwheel extension shaft meshing with a ring gear fastened to the inside of the rotating hood structure. A set of collector rings mounted on the vertical centre column supplies current to the five roll motors, the spray pump and the spray valves. The timers are located in the main control room on the chiller control desk. The latter also carries the turntable rotation controller and the hydraulic jib crane controls.

The final laydown station consists of a simple turntable carrying three stacking pegs. The pegs, designed to receive five completed wheels, have their upper end shaped to suit a set of automatic tongs fitted to the overhead crane used for removal of the completed stacks of wheels to the cooling beds.

Motor Room—Hydraulic Plant

The main pumps and accumulators for the press hydraulics, which are designed to operate up to a maximum working pressure of 2,200 lb./sq. in., are arranged to form two independent systems with separate control gear. The large system comprises five 68 cu. ft. capacity strip-wound air vessels and one 68 cu. ft. capacity strip-wound water vessel. The small system comprises two 68 cu. ft. strip-wound air vessels and one 25 cu. ft. solid-forged water vessel. The ratio of air to water volume is such that a pressure drop of 10% is allowed over the normal operating levels. The 68 cu. ft. vessels are made from welded plates covered by four layers of special section rolled steel strip. A 3-stage Hamworthy air compressor of 18 cu. ft. / min. capacity and $14\frac{1}{2}$ h.p. is used for charging the air vessels. The water and air vessels are connected to mercury level control pots, of Vickers–Armstrong design, for control of the pumps and accumulator auto-stop valves. The pumps are unloaded by lifting the suction valves at pre-determined water level. Contacts in the mercury pots shut down the pumps at extra high level to avoid overfilling. Should the pumps fail to meet demands on the systems, further contacts are arranged which close the accumulator auto-stop valves thus isolating the water vessels from the press supply lines.

The large accumulator system is fed by two three-throw Vickers–Armstrong pumps of Elswick design each having a capacity of 400 gal./min. at 2,200 lb./sq. in. The rams are $4\frac{5}{8}$ in. diameter $\times$ 21 in. stroke, and the crankshaft speed 110 r.p.m. Each pump is driven through single reduction double helical gears by a Metropolitan–Vickers 6,600-volt slip-ring motor of 750 B.H.P. at 740 r.p.m.

The small accumulator system is fed by a Davy three-throw pump of 160 gal./min. capacity at 2,200 lb./sq. in. having rams $3\frac{1}{4}$ in. diameter $\times$ 12 in. stroke, and a crankshaft speed of 150 r.p.m. The pump is driven through a single reduction double-helical gear by a Metropolitan–Vickers 6,600-volt slip-ring motor developing 275 h.p. at 740 r.p.m.

It is possible by means of the independent accumulator systems to operate the 8,500-ton press at lower pressure than the punching and dishing presses, and thus to consume less power in the forging of smaller products. The accumulators can be connected together to operate as a combined system by equalising the air pressures and opening a stop valve in the water lines. The pumps and accumulators are housed in the motor room and the valves are arranged under the vessels in a basement which extends into the main pipe subways and the press foundations.

Removable Element Hook Pins

Birlec Ltd. announce the development of an improved, removable, hook pin for supporting heating elements. This consists of a fluted shaft, generally cruciform in section, and pointed at one end. At the other end, set at an angle, is the horizontal ledge on which the element tape rests, together with a turned-over lip preventing the tape slipping off. Also set at an angle to the shaft, a vertical stop prevents the pin turning in its hole, any rotary movement being restricted by this stop digging into the brickwork. The pin is set diagonally in holes in the furnace wall and can be easily replaced; it supersedes the conventional design, which is built permanently into the brickwork, and thus obviates the expense and long furnace shutdown formerly necessary for the replacement of pins.

Foseco Laboratory Extensions

Building commenced recently on an extension to the laboratory of Foundry Services Ltd., Nechells, Birmingham. The need for increased space is due to greatly expanding business, both at home and overseas, resulting in greatly increased calls on the Foseco Technical Service.

Low Frequency Induction Heater

Equipment for Heating Stock Prior to Hot Working

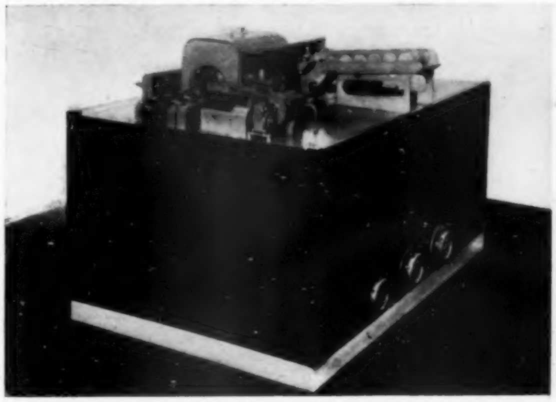
THE use of induction methods of heating stock prior to forging or extruding has grown appreciably during the last few years. Considerably less floor space is needed than is the case when fuel-fired or resistance furnaces are used, and as all the heat loss is carried away by the cooling water, working conditions are excellent. A further advantage is that furnace warm-up is eliminated, the first heated billet being delivered in a few minutes.

The Loewy Engineering Co. Ltd. is now building, under exclusive licence from the Magnethermic Corporation, Youngstown, Ohio, "Magnethermic" 50-cycle induction heaters which can be supplied completely assembled ready for connection to a 3-phase 50-cycle supply and a water supply.

The heater consists of an induction coil in which the billets are placed for heating. This coil carries a high current, which induces in the billet eddy currents whereby it is heated. Power is supplied to the coil through step-down transformers, and tap switches are provided to adjust the coil voltage. The water-cooled furnace transformers, the hand tap switches and the auxiliary controls are housed in a steel-framed casing with sheet steel panels, and the water-cooled heating coil, charging and discharging mechanisms, and thermocouple are mounted on top of the casing. The tap switches are interlocked with the access doors and the main circuit breakers. Air break contactors are provided for automatically switching current on or off the heating coil. A water pump is mounted in the heater casing for ensuring that cooling water flows at the necessary pressure to the transformers and the coil. The pump motor and heater control gear are electrically interlocked, so that the heater cannot be operated unless cooling water flows through the heating coil and transformers.

The heater is designed to heat one, two or three billets simultaneously, depending on the billet length, but with only one billet being ejected at a time. Billets are automatically fed into and ejected from the heating coil by a motor driven pusher controlled by push buttons and limit switches set to suit the length of billet. A short roller table, motor driven, is provided to carry the billet clear of the coil once the pusher has started to eject it. A number of billets are loaded on to the inclined loading ramp at the rear of the coil and from this point they are automatically fed into the heater and brought up to temperature. Alternatively, the heaters can be supplied with a pneumatic billet pusher.

Billet temperature is measured by a pair of chromel-alumel thermocouple points housed in the retractable stop against which the billet rests during the heating period. The thermocouple is connected to a 12 -in. high speed indicating and recording temperature controller, which is arranged for separate mounting, and is equipped with micro switches which cut off the power immediately the billet reaches the temperature for which the controller is set, and re-apply the power if the heated billet drops in temperature by 10° C. In operation, the rate of heating is adjusted so that the billets are brought up to temperature shortly before discharge from the coil should take place. During heating the outer skin of the billet is brought up to temperature quicker than the inside, and the core and skin equalise in temperature after the power is cut off. For example, a 6 in. billet with 55° C. difference equalises in about 30 seconds. Successive billets, if taken immediately they come up to the desired temperature, would be consistent in temperature within ± 3° C. The heat pattern in the coil can be varied by means of the tap switches.



50-cycle induction heater with thermocouple in contact with billet during heating.

The insulation of the water cooled heating coil is protected from internal abrasion by a stainless sheet steel liner. In the bottom of the coil is an additional stainless steel piece on which the billet slides. This piece prevents wear on the coil liner and is easily renewable. Spare billet slides are provided so that if one wears or distorts excessively, the spare can be substituted while cleaning and straightening the other.

Each heating coil is made to take one specific nominal size of billet with a tolerance of about ± $\frac{1}{8}$ in., which allows for irregularities in billet surface. With smooth billets the tolerance is larger, and about ± $\frac{1}{4}$ in. Any diminution in heating efficiency due to using a smaller diameter billet can be offset by applying a slightly higher voltage to the coil by adjusting the tap switches. When the size of billet to be heated is altered, the heating coil units can be easily interchanged in about 45 minutes, since the only parts to be disconnected and reconnected are the four bolted bus bar joints and the two cooling water hoses. Coils are cool enough to handle as soon as the power is switched off; the outer parts of the coil, including the outer parts of the turns, do not rise above room temperature.

The power rating refers to the power consumption during the actual heating time. If smaller quantities of billets are heated than the rated capacity of the heater, the rate of heating can be reduced by varying the tappings of the transformers. Since the heating

time is very short it is possible to employ the "Magnethermic" heater for infrequent demand of billets without constantly applied current or waste of power. Furthermore, the heaters can be put into operation in a matter of minutes as no warm-up time is required, so that during a temporary press shut down the heaters can be switched off completely and no power is consumed. This also eliminates the labour normally required in preparing a conventional type furnace before a working shift begins. Changes from one alloy to another can quickly be made without waste of heat or time.

The inherent power factor of these heaters is 0·44 and this can be corrected to any desired power factor with capacitors. The capacitor bank is external to the furnace and can be mounted in any convenient position to suit site conditions. Capacitor banks for alternative power factor correction to 0·85, 0·90 or 0·95 are available.

The "Magnethermic" heater will heat 8 to 9 lb. of light alloy billets to 450° C. per kilowatt-hour, and the heat efficiency is 45–50%.

The indicating lamps, push buttons and control circuit breaker are mounted in an operator's control panel which can be mounted conveniently for operation by the press operator, and the whole sequence of operations can be made fully automatic.

Air-Blast Circuit-Breakers for Arc Furnace Control

At the beginning of the last war a large number of arc-furnaces were built for the production of special steels for armaments, and at that time it was established that the standard oil circuit-breaker was unsuitable for the heavy duty of controlling the H.V. supply to the furnace transformer, which normally involved about 500 operations per week, breaking currents ranging from the transformer magnetising current up to fairly heavy fault currents.

Very careful and assiduous maintenance was required on the oil circuit-breaker equipments every week, necessitating changing or filtering the oil and dressing the contacts. A number of these equipments were supplied with specially strengthened bearings and other moving parts and in some cases special oil filtering equipment was actually fitted to the breakers. Some accidents occurred where the maintenance had not been quite as good as was required.

Contacts of a 6·6 kV, 150 MVA, 800 amp. air-blast circuit-breaker after 26,000 operations.

During this time the air-blast circuit-breaker was being developed and it became obvious that this type of breaker would be far superior to the oil breaker on this heavy duty, due to the fact that the arc is drawn in air, a fresh supply of which is provided for each break operation, and to the advantages of pneumatic control, namely, flexibility and lightness of moving parts.

A number of air-blast circuit-breakers were supplied during the war and in the following years, which have given consistent service on this arduous duty at voltages ranging from 3·3 kV to 33 kV. Maintenance was reduced to a fraction of that required on the oil breakers, and experience on an "English Electric" air-blast circuit-breaker equipment installed at the Brymbo Steel Works at Brymbo near Wrexham brings out very clearly the superiority of the air-blast circuit-breaker for this application.

The breaker rated at 6·6 kV, 150 MVA, 800 amps., was the first commercial air-blast circuit-breaker to be installed by a British manufacturer and went into commission early in 1940. After the initial minor troubles, the breaker has given trouble-free service ever since, except for a period of about two years just after the war when the furnace was out of commission. After continuous operation for 13 months, involving about 26,000 operations, the breaker has recently been stripped down. Apart from the weekly external inspection and draining the water from the compressor receiver, no maintenance had been carried out.

The illustration shows the contacts on the three phases just after the breaker was stripped down, and the wear is so slight they are being refitted for further use. The pneumatic equipment, including the control valves, blast valves and compressor equipment were examined. All parts were found to be in perfect order, and the whole equipment is being returned for service without any replacement being necessary. It is interesting to note that the pneumatic equipment and the compressors require as little maintenance as the air-blast circuit-breakers themselves. Such a record could only be achieved with an air-blast circuit-breaker, and clearly demonstrates the superiority of this type of breaker for arc-furnace control.

The only advantage which an oil-breaker appears to have over an air-blast circuit-breaker is that its initial cost is generally less than that of the air-blast circuit-breaker and its compressor equipment. In fact, however, this extra cost is small in proportion to the total cost of the furnace installation, and the extra expenditure on air-blast equipment is more than compensated during the life of the furnace by the saving in maintenance and repair.

Review of Recent Developments in Electroplating

By H. Silman, B.Sc., F.R.I.C., F.I.M., A.M.I.Chem.E., A.I.Mech.E.

In the last year or two there has been a number of interesting developments in both methods of electroplating and the equipment used. This review, which discusses a selection of these, reflects also the efforts made during that period to develop substitutes for nickel as an undercoat for chromium.

THE main problem which has faced the metal finishing industries in the last year or two has been the continuing shortage of materials, of which the most important has been nickel. It is, therefore, not surprising that much activity has been concentrated on finding substitutes for this metal, which has hitherto been the almost universal undercoat for chromium. Although nickel will continue, in all probability, to be the main metal employed for this purpose, a number of the substitutes which have been introduced are certain to find some application, especially for decorative uses under conditions which are relatively mild.

In spite of shortages, progress has been very rapid in all branches of the field, particularly on the plant side. The introduction of new materials and new mechanical developments is having a revolutionary effect on plating methods in current use, and this progress is likely to be maintained.

Substitutes for Nickel Plate

White Brass.

The use of a deposit of white brass to replace nickel under chromium has made a fair degree of headway in the United States, but less so in this country. A cyanide solution is used, with or without addition agents which serve primarily to brighten the deposit, although they also improve the throwing power of the solution to some extent. The alloy which is deposited may contain from 10% to 25% of copper.

The cyanide bath employed has a high caustic soda content; it has been found that the concentration of the constituents is of less importance than the ratios of Zn/Cu, NaCN/Cu and NaOH/Cu, which should be, respectively, 2·7, 6·9 and 5·0[1].

The anodes employed are of copper and zinc in the proportions of 80% zinc to 20% copper, and the bath temperature is around 100° F. The optimum thickness of the deposit is of the order of 0·0002 in. to 0·0003 in.; lighter deposits, particularly on steel, are not of sufficient durability, whilst heavier deposits tend to craze.

The fully bright white brass coatings take chromium very well, but plating of the dull deposits, after polishing, is more difficult. The reason is probably that the polishing compounds adversely affect the ability of the deposit to accept the chromium plating.

White brass can be plated in conventional equipment, including barrels, and the deposits adhere well to steel, brass and zinc-base alloys. The deposit is very ductile, but for outdoor exposure the coating has not proved adequate in its protective properties, white corrosion products developing relatively quickly, with the consequent failure of the chromium coating. This happens more quickly on steel, as the deposit is anodic to it; copper undercoats are not very helpful in improving corrosion-resistance.

Tin/Nickel Alloy Plate.

An interesting development has been the introduction of an alloy deposit of tin and nickel consisting of an intermetallic compound which is fairly hard and of a pleasing appearance. The colour is a little more yellow than chromium and in some lights appears to have a purplish tint; the plate resists tarnishing well. The electrolyte used contains stannous chloride, and sodium fluoride, the preferred composition being: stannous chloride 50 g./lit.; nickel chloride 300 g./lit.; sodium fluoride 28 g./lit.; and ammonium bi-fluoride 35 g./lit.

The working temperature is 65° C. and with a current density of 25 amps/sq. ft. a bright plate is obtained over irregular objects at a current efficiency approaching 100%.

The composition of the deposit does not appear to depend greatly on variations in current density and temperature; even variations of 20% to 30% in the content of either metal in the electrolyte produce insignificant changes in the composition of the deposited alloy. The chief factor affecting the composition appears to be the total fluorine content of the electrolyte. The bright plating range is obtained at high rather than low temperatures, and the presence of hydrofluoric acid and an alkali metal ion (preferably sodium) is necessary for the best results.

The bath may be replenished either by using alloy anodes or by employing separate tin and nickel anodes in circuits with different applied voltages. With separate anodes, precautionary measures are necessary to minimise the effect of chemical reaction between the tin anodes and the electrolyte, which leads to the formation of a loosely adherent deposit on the tin anodes. This deposit consists mainly of tin and nickel with a nickel content of around 24%. Its presence does not greatly interfere with the cathode process or with the dissolution of the anodes, but it represents a wastage of tin and nickel and can be troublesome in practice. The best way of avoiding the formation of the deposit is by surrounding the tin anodes with bags made of several thicknesses of Terylene fabric. The tin anodes can then be operated at current densities of up to 50 amps./sq. ft. Dissolution of the nickel anodes presents no difficulties as sludge is not formed on them and it is not necessary to remove the anodes when the bath is idle. Anode current densities of up to 50 amps./sq. ft. can also be used with these, the efficiency being around 100%.

Impurities must be avoided in the bath. The upper limits for metallic impurities are given by Parkinson[2] as: iron 0·5 g./lit.; copper 0·2 g./lit.; antimony 0·4 g./lit.; zinc 1·5 g./lit.; and cadmium 1·5 g./lit.; lead must be kept below 25 parts per million. Copper is probably the commonest impurity, excessive quantities causing a serious fall in the brightness of the deposit.

This impurity can, however, be removed easily by treating the bath with nickel powder. Organic impurities are especially deleterious, leading to very serious pitting of the deposit, and can only be eliminated by filtration through activated carbon. The solution is operated in rubber-lined tanks ; the rubber employed must be of high quality and the presence of free sulphur and certain fillers must be avoided. Perspex, polythene and unplasticised polyvinyl chloride also resist the electrolyte, although the two latter materials are weak at the operating temperature.

On non-ferrous basis metals, a thickness of 0·0005 in. of tin-nickel appears to give reasonably good service, but on steel it is preferable to apply an undercoating of 0·0005 in. of copper if adequate durability is to be obtained. The composition of the deposits is always around 65% of tin and 35% nickel, and it appears to have a metastable nickel arsenide type structure similar to that of $\alpha-Ni_3Sn_2$.[3] The alloy deposit is stable up to 300° C. but decomposes on heating above this temperature.

Bronze Plating.

Bronze deposits containing a relatively high proportion of copper (85% to 90%) have been introduced commercially in this country as a substitute for nickel undercoats, and have proved suitable for direct chromium plating. A high degree of durability under conditions of atmospheric exposure has been claimed[4]. By the use of addition agents, a fully bright deposit which requires no buffing is obtainable. On steel, a deposit thickness of 0·001 in. of bronze is considered adequate for normal outdoor service.

The solution used is of the cyanide type and is best operated in a mild steel tank with alloy anodes. It can be plated directly on to steel, brass and zinc base alloy die-castings. According to Duc[5], the free cyanide and free caustic soda contents of the bath should be closely controlled at 17 g./lit. and 12 g./lit. respectively, when little or no metallic additions should be needed. The temperature is 145° to 160° F., current densities of about 25 amps./sq. ft. being recommended ; the throwing power is as good as that of a stannate tin solution. Continuous filtration is highly desirable. Exposure tests have given good results with this type of combination. Silman[6] has reviewed the corrosion resistance of chromium deposits applied under various conditions. In particular, fully bright chromium deposits can be plated at reasonable efficiencies from a bath containing 250 g./lit. of chromic acid and 2·5 g./lit. of sulphuric acid at temperatures of 140° to 150° F., and current densities of 400 to 500 amps./sq. ft. Such deposits have been found to have outstanding corrosion-resistance, particularly when applied directly on to bronze plating on brass or steel.

Periodic Reverse Current Plating

A good deal of interest has been shown in the methods of plating with periodic reversal of current. The process has been chiefly used with cyanide copper electrolytes, the object being to increase the rate of deposition and, at the same time, improve the levelling properties of the deposit, thus enabling basis metals with a low initial finish to be employed[7] ; under suitable conditions, fully bright deposits can be obtained. In principle, periodic reversal of current involves making the article anodic for short periods during the plating operation. In this way the tendency for incipient nodule formation in the deposit is substantially reduced. This makes the process particularly applicable for the electro-deposition of heavy coatings. It is also claimed that there is an increase in throwing power with a faster rate of deposition, although the efficiency per cycle is lower. Anode corrosion is uniform and little trouble is experienced with the formation of anode films. In order to determine the best cycle to be used, some tests are generally necessary. The most commonly used cycle is approximately 15 seconds plating followed by 5 seconds deplating. For increased levelling or exceptionally smooth deposits, P.R. cycles of the order of 20 seconds plating followed by 15 seconds de-plating, with very high current densities are employed. It is thus seen that where a high degree of brightness is required the deplating time has to be increased to a very substantial extent, which results in a much lower overall efficiency. For the best results, switching must be rapid with the P.R. process ; if the current does not build up to the full amount immediately after each reversal, efficiency values and plating speeds are reduced whilst inferior deposits may result. The factors affecting levelling have been reviewed by Jernstedt[8].

Courtesy of W. Canning and Co. Ltd.

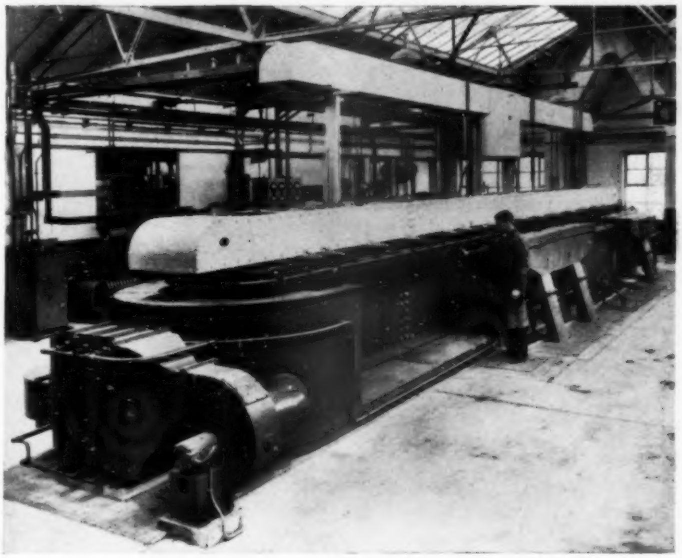
Fig. 1.—Chromium plating barrel.

The current may be reversed either by reversing the field excitation of a D.C. generator or by means of suitable contacts on the low voltage output of the source of D.C. current, which may be either a generator or a rectifier; the field reversal system is not recommended unless the reverse period is at

least 5 seconds. Arrangements are also needed to ensure that the work enters the bath whilst it is cathodic. Timers for use with P.R. equipment can be either of the electronic or mechanical types.

For the best results, certain inorganic addition agents are recommended, particularly zinc and thiocyanates[9]. The zinc is added in the proportions of 0·3 to 4·27 g./lit., with a preferred concentration of 0·75 g./lit., to the copper solution, which may be of the normal cyanide type, or alternatively, a Rochelle salt solution can be used. The zinc concentration is critical, as excessive quantities cause fogging of the deposit.

A suitable composition for introducing zinc and thiocyanates simultaneously consists of a mixture of 1 part by weight of zinc oxide, 2 parts of copper thiocyanate and 5 parts of an alkali metal cyanide. Addition of certain organic compounds, such as betaines, extends the bright plating range, but thiocyanates are preferred.



Courtesy of Electrochemical Engineering Co. Ltd.

Fig. 2.—Return-type automatic nickel and chromium plating plant.

P.R. Nickel Plating.

By the use of pulsed current plating, with a cycle in which the cathodic period is generally not more than 2 seconds in duration followed by an anodic period of at least 0·002 second, it is claimed that smooth and exceptionally pore-free nickel deposits can be obtained which are fully bright over a wide range of current densities up to 500 amps./sq. ft.[10].

Conventional nickel plating solutions are used with or without suitable addition agents, amongst those recommended being naphthalene 1,5 disulphonic acid and sodium lauryl sulphate. Using a Watts type of solution, a cathodic period of 0·2 second and an anodic period of 0·04 second gave smooth bright deposits at currents of up to 150 amps./sq. ft. The *p*H of the bath was 3·0 to 4·3 and the temperature 115° to 140° F. A cobalt-nickel bath gave similar results with a periodic reverse system of 1 second cathodic and 0·2 second anodic at current densities up to 150 amps./sq. ft. These deposits were much smoother and brighter than those obtainable by conventional methods. Even brighter deposits at higher current densities could be obtained by reducing the cathodic period to 0·2 second with an anodic period of 0·04 second.

Plating of Aluminium

In the field of plating of aluminium there has been a valuable addition to the literature in the form of a full and detailed description by Wallbank[11] of the Vogt Process, which is probably the most widely used commercial process in this country. By this method all aluminium alloys, except possibly those containing more than 3% of magnesium, can be successfully plated, as can assemblies containing other metals.

Essentially, the process consists in first electrolytically cleaning the articles in a caustic soda and cyanide cleaner, after which they are rinsed and etched in a strong solution of nitric and sulphuric acids. After electro-cleaning once more, they are thinly plated with zinc followed by brass and then nickel plated in a solution containing very little chloride, under conditions giving a soft unstressed deposit. Finally the articles are stoved for half an hour at 450° F., which improves the adhesion of the deposit. At the same time coatings showing defective adhesion will blister during this operation, so that those likely to give trouble can be rejected.

The essential feature of the process is the zinc plating operation which is carried out in a bath consisting of :—

Zinc Chloride	0·5 g./lit.
Sodium Cyanide ..	0·5 g./lit.
Caustic Soda	10·5 g./lit.

The bath is operated at room temperature, with a current density of 5 amps./sq. ft. ; the plating time is 20 seconds. No attempt is made to control the bath by analysis, the deposit being judged by its appearance, which should be a uniform bronze. If the deposit becomes a blue-black in colour it is too thick and the bath needs diluting.

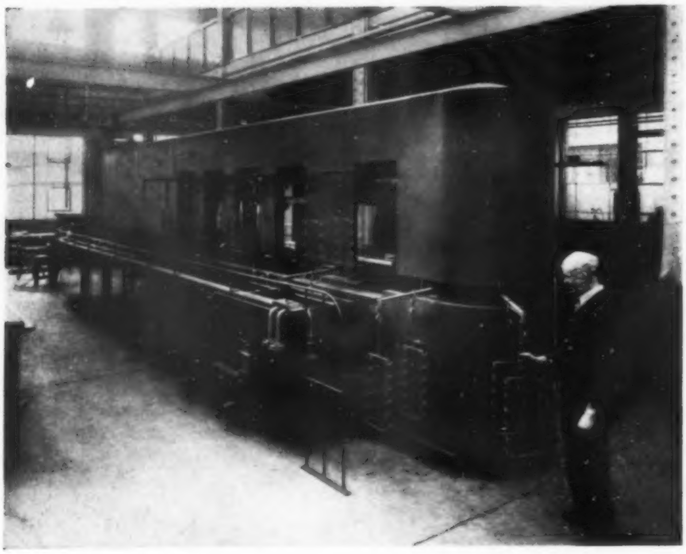
Anodising

Exceptionally hard anodic coatings on aluminium by anodising have been developed both in this country and in the U.S.A.[12] These oxide coatings are thicker and denser than the usual type of coating (up to 0·01 in. in thickness) and enable aluminium to be used when lightness and high resistance to wear are important. The process is presumably carried out by anodising at low temperatures in suitable electrolytes to minimise attack on the coating by the electrolyte. Refrigeration of the solution may be necessary at the high current densities used.

A similar British process is also now commercially available, and can be applied to alloys of high silicon or copper contents for which the American process is less satisfactory. The film is either grey or brown in appearance, is rather brittle and is sometimes crazed when it leaves the bath. Crazing also tends to develop if the coating is heated to temperatures in excess of 300° C.

Chromium Plating

The first major development in chromium plating for



Courtesy of W. Canning and Co. Ltd.

Fig. 3.—Return-type automatic plating plant for one operator.

many years has been the introduction of the Self-Regulating High Speed Chromium Plating Bath (known as the "S.R.H.S." Bath). This bath is of the low density type (200 to 300 g./lit.) and is characterised by the fact that the catalyst, which is of the silico-fluoride type, is of limited solubility and so automatically regulates its own concentration within the bath. High current efficiencies are claimed, i.e., from 4% to 10% higher than that obtained in the conventional solutions of similar concentration, depending on the conditions of plating. There is also said to be an improved ability to plate on passive nickel and relative freedom from iridescence due to thinly plated areas. The S.R.H.S. bath is coming into increasing use in the U.S.A. and to a lesser extent in this country.

The barrel plating of chromium, which is very difficult to carry out, is now a practical proposition as a result of the introduction of several new types of plant (Fig. 1).

Plating of Magnesium

A practical process for the plating of magnesium which has hitherto proved an exceptionally difficult metal to plate, has been described by deLong[13]. In this method the parts are degreased, pickled in a bath containing 20% of glacial acetic acid and 5% of sodium nitrate, and then immersed for 3 to 5 minutes in a solution containing tetrasodium pyrophosphate 120 g./lit., zinc sulphate 40 g./lit., potassium fluoride 10 g./lit. and potassium carbonate 5 g./lit. After this treatment they are rinsed and plated with copper from a Rochelle salt bath. Deposits of excellent adhesion and appearance are said to be obtained.

Electroplating Plant

The tendency in the industry has been increasingly in favour of mechanisation of plating both from the points of view of reducing labour and improving quality. In the automatic plating field, plants are being designed which are more compact and simple than hitherto, the return type of machine being especially favoured. This design enables a lengthy sequence of operations to be carried out automatically, loading and unloading being at the same position, so that the plating plant can be conveniently operated by one man. A further advantage is that in many cases the plating jigs can remain on the plant, so that the problem of jig storage is eliminated. To prevent the plating up of contacts whilst jigs are travelling round unloaded, a simple method of insulation can be devised so that contact is made with the cathode when the jig is hung on to the carrier one way but is insulated from it when turned around. Labour may be further reduced by the incorporation of a pneumatic mechanism on one type of plant which automatically transfers racks from a main conveyor to the plating plant conveyor. Figs. 2 and 3 show two of the most successful return-type automatic plants in general use in this country.

For special purposes, however, large plants are still advantageous; an installation which is probably one of the largest in the world for the plating of steel window frames with zinc has been described by Smart[15].

Filtration Plant

Filtration equipment for plating solutions continues to leave much to be desired, although some progress is being made. Most existing types of plant are either inadequate so far as the quality of the filtrate is concerned, or they require a great deal of labour for cleaning and maintenance. An interesting recent unit, shown in Fig. 4, however, attempts to overcome these difficulties. Filtration is carried out by pumping the solution through filter sleeves on wire frames; the sleeves are of felt or cotton covered with nylon. The purpose of the nylon is to facilitate sludge removal when a back water pressure is applied to the unit, since the sludge does not adhere strongly to the nylon fabric. The paper pad type of filter which is widely used, is very effective so far as removal of solid matter from the plating bath is concerned, but cleaning of the filter is laborious, whilst the cost of replacing the pads is high.

The older filter press is still probably more widely used than any other type and has been considerably improved in recent years by the provision of more rapid methods for dismantling and assembling, and by making the plates of hard rubber. Filter cloths of woven plastic materials are also available for dealing with difficult solutions.

Considerable improvements have been made in the manufacture of pumps. The LaBour and Mono pumps are well established types in the industry; the use of rubber and stainless steel combinations enable even the high-chloride nickel solution to be successfully handled with little trouble. Almost any metallic material will, however, be attacked if it becomes anodic in acid solutions, and an important factor in obtaining trouble-free operation is, therefore, the location of the pump, which must be installed in such a way as to avoid bipolar effects. A great deal of study has gone into the question of devising means of preventing this type of attack and a considerable degree of success has been achieved.

In the operation of continuous filtration plants it is advisable that the rate of flow of the filtrate should be known. In many plants the solution flows through a closed system, which makes it difficult to determine this without adequate instrumentation. Rotameters have been used for this purpose with considerable success, but there is a need for a cheaper type of rate of flow gauge.

Plastics in Plating

The use of plastics in plating processes has made tremendous strides in the past year or two, the chief applications being for plating racks, for tank linings, and for fume ducting. A completely self-contained bright-dipping cabinet, wholly constructed of rigid polyvinyl chloride, now on the market, improves the conditions under which this operation is carried out. The system used involves the extraction and neutralisation of the fumes evolved by means of ammonia, followed by absorption in activated carbon.

Tank linings are increasingly being made of rigid polyvinyl chloride, and for certain applications they are superior to rubber. Thus, oxidising acids, such as chromic acid, can be contained in polyvinyl chloride-lined tanks. The chief limitation is temperature, however, such linings not being satisfactory where temperatures exceed about 50° to 60° C. Plastic linings can be loose fitting in a steel tank or bonded to it. The latter procedure is more satisfactory, but the unplasticised material (which is more resistant chemically and withstands higher temperatures than the plasticised polyvinyl chloride) tends to crack due to temperature changes when bonded directly to steel by means of adhesives, owing to its high coefficient of expansion.

A recently introduced system, however, makes use of a double layer of the plastic, a plasticised sheet being located between the metal and the rigid lining to take up movement due to temperature changes. The result is that a satisfactory rigid polyvinyl chloride lining can then be produced on steel tanks.

Fabrication of the tank linings is effected by means of welding, preferably using hot air or nitrogen from a suitable gun. For the best results, the position of the welds must be carefully studied. Exhaust and fume ducts are also being made by these methods with great success, painting and maintenance being almost eliminated. Polyvinyl chloride impeller pumps are available and are being used increasingly for handling acids where high temperatures are not involved.

Rigid polyvinyl chloride can be successfully extruded, and pipes up to 6 in. in diameter are being produced commercially, whilst large units can be fabricated by welding. The high co-efficient of thermal expansion of the material, however, again demands that consideration be given to avoiding cracking by allowing for this characteristic.

Polythene tank linings are employed with some success, but the material is usually flame sprayed on to the sheet as there is no satisfactory method of bonding a polythene lining. Welded polythene duct-work is also being increasingly specified. Polythene can likewise be welded by the hot torch process using polythene filler rods. With this material, an inert gas such as nitrogen is essential. Effluent and drainage pumps, agitation coils, storage bottles for acids, buckets, jugs, etc. are now being manufactured, with considerable economies. The material is extremely resistant to acids and alkalis

Courtesy of W. Canning and Co. Ltd.

Fig. 4.—Totally enclosed filter unit for plating solutions.

and has a tensile strength of the order of 1,500 lb./sq. in. with excellent extrusion properties. Manhole linings and catchpits are being made of polythene and acid-resistant pipes of the material are commonly buried underground for taking away effluents.

Plating Rack Coatings

Plating racks are now almost invariably insulated to conserve metal and to produce better and more uniform deposits. In this country practically all other types of insulation have been superseded by polythene, or resin dispersion coatings. Polythene is generally applied by heating the rack and sprinkling on the polythene powder, which is subsequently fused by heat.

The resin dispersion types are high-solid dispersions of vinyl resins in substantially non-volatile plasticisers, with or without the presence of organic solvents. The plastisols do not contain volatile solvents and are, in effect, 100% solids. In an organosol, the liquid phase contains both plasticisers and solvents. The advantage of the organosols is that the viscosity of the latter is more readily controlled, but the presence of a volatile content is a disadvantage in other respects.

Plastisols are the most widely used materials. The method of application consists in dipping the racks into the plastisol, avoiding the formation of bubbles, followed by stoving at 350° to 375° F. after draining. Plastisols do not generally adhere well to metal, so that a stoved primary coat is often applied before the racks are dipped into the coating material.

During stoving, the plasticisers, which at room temperature have only a slight solvating action on the resin particles, become more active until the resin becomes freely soluble in the plasticiser. The homogenous liquid produced is, however, in the form of a

gel which does not flow to any appreciable extent, and on cooling the familiar rubber-like covering is left.

These coatings are very durable and have now become widely employed. To some extent, plastisols are also finding application for the treatment of duct-work, and even dipping baskets and similar items of equipment. The subject has been reviewed by Williams[16].

Electropolishing and Chemical Polishing

Considerable interest is still being shown in electropolishing, and a large number of patents of various kinds have been taken out. The processes, have not, so far, developed well commercially on metals other than stainless iron and steel, where the value of electropolishing is fully established. Considerable polishing economies are effected and improved corrosion-resistance is obtained. Non-electrolytic polishing methods have also achieved some degree of success, and processes are available for the treatment of brass, copper, aluminium and steel. Chemical smoothing of steel has been described by Marshall[17] who uses a solution consisting of:—

Oxalic Acid	25 g./lit.
Hydrogen Peroxide	13 g./lit.
Sulphuric Acid ..	0·1 g./lit.

The process has received practical application on a considerable scale. A feature of the treatment is the fact that there appears to be a film left on the surface of the metal which has appreciable corrosion resistance. The evidence indicates that the film consists of a thin tenacious layer of oxide of an estimated thickness of 60Å[18].

Effluent Disposal

The constant demands of local authorities for improvement in the quality of effluents to avoid pollution of rivers has resulted in a good deal of research into methods for dealing with plating wastes. The most difficult materials to be dealt with are cyanides and chromates, the simple neutralising chamber containing lime and iron now being considered inadequate for this purpose. Treatment by milk of lime and ferrous sulphate will deal with chromium wastes if properly carried out[19], but cyanides present a more difficult problem[20]. In the United States oxidation of the cyanide by available chlorine is the most widely used method[21], whilst another method consists in volatilising the cyanide as hydro-cyanic acid with appropriate precautions[22].

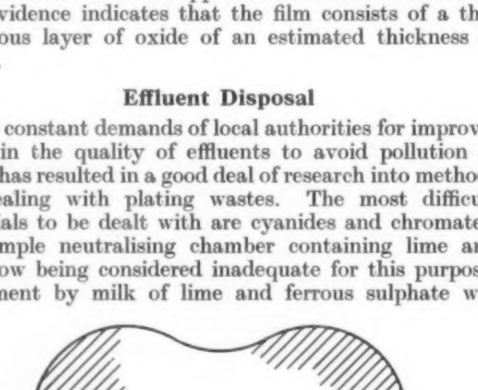


Fig. 5.—Schematic view of an adhesion test specimen with electroformed nodules for gripping the elect o-deposits.

To conserve acid in pickling, and also to reduce the amount of pickling wastes, acid regeneration systems are now being employed on an extensive scale. In the case of pickling plants for steel, the waste acid liquors containing iron equivalent to 85% solubility are pumped into the crystalliser through which refrigerated or deep well water is circulated, whilst the acid is rapidly stirred. By lowering the temperature of 10% sulphuric acid to 20° C. from 60° C. the iron content of the solution can be reduced to about 6% in 2 hours. The ferrous sulphate crystals are removed, washed and dried and the regenerated acid returned for further use[23].

Copper is also being recovered electrolytically on an increasing scale from the acid residue obtained in the pickling of copper bar and rod.

Testing

A means of obtaining absolute values for the adhesion of thin deposits has been developed which appears to offer advantages over previous methods, in that it is the only generally available method permitting the measurement of the adhesion of coatings of commercial thickness[24]. In principle, the method consists in plating a cobalt nodule on to the deposit to be tested through a suitable plastic washer and then pulling the nodule off by a standard tensile testing apparatus. No machining operation is involved. A development of the nodule method which has been described more recently[25] consists in electroforming identical concentric cylindrical nodules on both ends of the electroplated base metal which gives a miniature tensile specimen with the electrodeposited base metal sandwiched between the two nodules (Fig. 5).

Cleaners

The testing and evaluation of cleaners have been the subject of a good deal of work. The definition of a "cleaning efficiency index" as a quantitative figure was first described in 1946, the method used being to subject a metal panel which has been contaminated by a standard procedure to the cleaning cycle, rinse in running water for 5 minutes at 50° C., to eliminate water breaks, and then spray with clean water fiom a fine atomiser. Any residual oily areas are shown up as a pattern, and their size can be estimated. Further work[27] has, however, shown that the oil film, which is initially continuous, shrinks and yields many discontinuities, in which no oil is evident. Those areas that are still covered with oil, however, have thicker films than they had originally. Finally, the oil assumes a spherical form with minimum attachment and is then removed. The water spray pattern apparently shows the area from which the oil has been removed either temporarily by shrinkage or permanently by physical removal.

In applying these tests, uniform metal surfaces should be used and care taken to rinse off the last traces of surface-active agents. Also, loosely adherent oil picked up from the surface of rinse waters should not be allowed to dry on the metal.

A method of testing cleaners using radioactive stearic acid has been described[28]. Tests showed that distinct

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How Powder Metallurgy is Progressing

By H. W. Greenwood*

Developments in powder metallurgy are proceeding apace and it is becoming increasingly difficult to keep in touch with them all. In this article the author discusses a number of the highlights of this progress, dividing his attention among metal powders, pressing and sintering, bearings, high temperature alloys and magnetic materials.

It is interesting to reflect that the first Symposium on Powder Metallurgy to be held in this country took place just over five years ago. This year there were congresses or symposia in which powder metallurgy was either the principal or a major theme on four occasions between the months of April and June. The first one in U.S.A. was the 8th Annual Meeting of the Metal Powder Association of U.S.A. held in Chicago (April 29th and 30th). There were two in Sweden, namely the 4th International Mechanical Engineering Congress in Stockholm (June 4th to 10th) at which nine papers on powder metallurgy were presented, aggregating some 130 pages of printed matter, and the International Symposium on the Reactivity of Solids at Gothenburg (June 9th to 13th) at which thirteen important papers on powder metallurgy were presented. Finally, there was the 1st Plansee Seminar on Powder Metallurgy held at Reutte-Tyrol (June 22nd to 26th) at which twenty-eight papers were presented. Earlier in the previous year there was the very important Symposium on High Temperature Steels and Alloys for Gas Turbines held in London (February 21st and 22nd), in which Group VII—Special Blade Materials—carried a most important paper on sintered alloys together with others on sintered ceramics and on sweat-cooling.

Powder metallurgy is occupying more and more space in technical journals and the volume of literature occupied by it increases almost daily. As a result it becomes increasingly difficult to keep track of the latest developments, and almost impossible to maintain that overall view that misses no important contribution to the subject. The volume of contributions also renders difficult any résumé of progress that can claim to be complete as well as comprehensive. All that one can do is to choose the matters that appear to be of real importance and value and hope that nothing of outstanding moment has been overlooked. As an illustration of the volume of material extant, mention can be made of the third volume of Goetzel's "Treatise on Powder Metallurgy" published this year, which comprises a classified and annotated bibliography. It contains 5,535 references to literature and 6,330 references to patents, and that may remind us that one of the outstanding items in the progress of powder metallurgy has been the publication of this monumental work, which now supplies a full treatise on the subject, with a bibliography that will prove invaluable to every worker in this field.

The Manufacture of Metal Powders

Of all the metal powders in use to-day, iron powder is the most important and will become increasingly so in powder metallurgy. In this country, although small quantities of carbonyl iron and electrolytic iron powders

High precision iron parts produced by pre-pressing, pre-sintering, coining and final sintering. Swedish products. (¼ natural size.)

are used, as well as some cast iron, the powder in predominant use is the Swedish sponge iron powder. A good account of the manufacture of this powder, which is covered by British Patent 675,350, appeared in *The Chemical Age*[1]. The largest producers of Swedish sponge iron powder are Hoganas Billesholms A.B., and they are constantly studying methods of production and of purification with a view to enhancing the properties and the purity of their product. The presence of silica in such a powder is a drawback in powder metallurgy because of the excess wear on dies and punches which it causes. A process has been devised by which treatment with a 2% solution of hydrofluoric acid for from 1 to 3 hours, followed by washing and drying of the powder in hydrogen at a temperature of 300° to 400° C. reduces the silica content from 0·3% to 0·05%[2].

The tremendous growth in the use of iron powders in the last year or so is due very largely to the adoption of various techniques that have overcome the defects from which the earlier products of iron powder metallurgy suffered. The commonest charge was that iron parts had poor strength due to high porosity. This could be overcome by the use of higher pressures during pressing, by the use of finer particled powders, coining, or even hot pressing, but these additions to the straightforward pressing and sintering added inordinately to the cost, and so the process became uneconomical and non-competitive. The first step in overcoming the difficulty was the introduction of infiltration, by which the pores in a porous compact were infiltrated with a lower melting point metal or alloy, such as copper for iron compacts, so that the initial porosity was eliminated, the pores being filled by the molten metal under the influence of capillarity, or because a vacuum was used; in the latter case the process was called impregnation.[3]

* Powder Metallurgy Ltd., London.

Additional advantages were conferred on this procedure by the use of precipitation hardening alloys as infiltrants. In this way, quite excellent tensile strengths were obtained and these infiltrated parts found application in many fields, not least that of compressor blades for gas turbines. The use of precipitation hardening alloys stimulated research into the control of the hardening processes by the addition of small quantities of elements which could accelerate or retard the hardening processes. This work is still in progress, but has already provided data whereby not only is control greatly facilitated but the physical properties are notably enhanced. The infiltration technique is now in use not only as a means of providing high density, non-porous parts, as by infiltrating iron parts, but is finding much wider application in the production of special hard alloys in which a skeleton of carbide is infiltrated by another carbide or other very hard compound.

The expanding applications of powder metallurgy have stimulated demands for a much wider variety of powders than was used until recently. As a result, novel methods of production have been successfully used, as for example the reduction of chromium oxide by lithium hydride[4] in which the two powders are pressed and the pressings heated under vacuum to 800° to 900° C. and, after cooling, leached in water to give a substantially hydrogen free chromium powder and lithium hydroxide. The advantage of using lithium hydride is that the reduction products are easily removed from the crucible, whereas when other reactants, such as lime, are used, the product of the reaction has to be chiselled out of the crucible.

Flake metal powders are used in a number of directions nowadays and their production calls for many methods. The older flake powders were produced by pounding turnings under a battery of hammers, or in an eddy mill. In addition to these methods, electrolytic processes are used ; one ingenious way of making flake nickel powder comprises a special automatic plating equipment which deposits alternately a nickel coating 0·00004 in. thick and a separating flash copper coating. In 3½ hours, 262 platings are completed. The sheets are cut into strips $\frac{1}{16}$ in. wide and again cut into $\frac{1}{16}$ in. squares, before being placed in weak acid solution which dissolves the copper and does not affect the nickel. The latter is centrifuged and washed, centrifuged and dried and then used as required[5].

Stainless steel powders, particularly in the production of filters for corrosive liquids have been produced in this country as well as in the United States. A particularly interesting production is that by B.S.A. comprising a powder having particles of sharp angular form and made by electrolytic intergranular corrosion of stainless steel[6]. Standard engineering filters have been produced having a total effective area of 9 sq. ft., and laboratory apparatus such as filter crucibles, Buchner funnels and other pieces of chemical apparatus is envisaged. Atomised stainless steel powders having spherical particles have also been produced in this country. An interesting and novel powder product is SAP sintered aluminium powder[7]. This is an aluminium powder admixed with some 10% alumina and compacted with a cellulose gum binder. On sintering after pressing, it provides remarkably high physical properties at relatively elevated temperatures, shows excellent thermal shock resistance and opens up a really remarkable range of possibilities. Distantly allied to SAP is the extremely interesting ceramic tool material introduced by B.S.A. and known as B.S.A. Sintox[8]. This is a sintered alumina range of standard turning and boring tools, produced by Lodge Plugs Ltd. and introduced by B.S.A. Tools Ltd. of Birmingham, which has shown remarkable resistance to abrasion and high cutting and boring speeds. This may well be the harbinger of a series of abrasion and heat resistant sintered oxides having good resistance to thermal shock, very high hardness and very high melting points.

Before leaving the general subject of the manufacture and properties of metal powders, a word or two must be said about coated powder particles and coated surfaces, a subject most conveniently handled at this point. The coating of iron powders with copper, especially those of hard angular character is now quite well known. An application of this has been the use of additions of up to 20% of copper powder to iron powders to increase the strength and improve the properties of sintered compacts. Powders so treated sometimes swell during sintering while others may shrink. This is an inconvenience and it has been found (B.P.680,275) that such swelling or shrinking can be very notably modified by the addition of small quantities of tungsten, either as the metal powder or as a tungsten compound such as tungstic acid. The decrease in swelling is accompanied by an improvement in tensile strength. In actual practice between 5% and 10% of copper is used and the nature of the results can be gathered from the figures in Table I which shows results obtained with pressures of 5 (metric) tons/sq. cm. and sintering for 60 minutes at 1,150° C. in a hydrogen atmosphere ; the parts were cylinders.

TABLE I.—EFFECT OF ADDITIONS OF COPPER AND TUNGSTIC OXIDE ON THE PROPERTIES OF SINTERED IRON PARTS.

Composition of Mixture	Alteration in Diameter—%	Tensile Strength —kg./sq. mm.
92·5g Fe + 7·5g Cu	+1·93	25·9
92·5g Fe + 7·5g Cu + 0·5g WO_3	+0·44	35·8
92·5g Fe + 7·5g Cu + 1·0g WO_3	+0·13	39·5
92·5g Fe + 7·5g Cu + 2·0g WO_3	−0·19	43·0
92·5g Fe + 7·5g Cu + 3·0g WO_3	−0·30	44·1

This procedure would appear capable of expansion in several directions. The addition of 0·5g. of chromic acid to the mixture somewhat enhances the effect in reducing the swelling of the compacts while hardly affecting the tensile strength. This example has been quoted at some length because it is typical of much work that is bearing fruit in these days, in which additions of quite small quantities of material are effecting changes and easing control capable of facilitating processes, increasing their economic value, rendering control more exact, and producing an all-round better article. Another use of coating is as a protection against oxidation at high temperature and molybdenum provides a good example ; trials with molybdenum alloys have given superior results. The metal itself has been given a siliconised coating, and has thus been able to withstand temperatures of the order of 1,850° C. for extended periods. Using nickel or Inconel cladding and suitable edge protection, wrought parts can be used in air at from 980° to 1,090° C.

Pressing and Sintering

As might be expected, the all-over acceleration in rates of production to-day finds examples in powder metallurgy as elsewhere. In pressing, this has been shown by the tendency to speed up the operation of feeding powder to the press, which in turn has led to consideration of

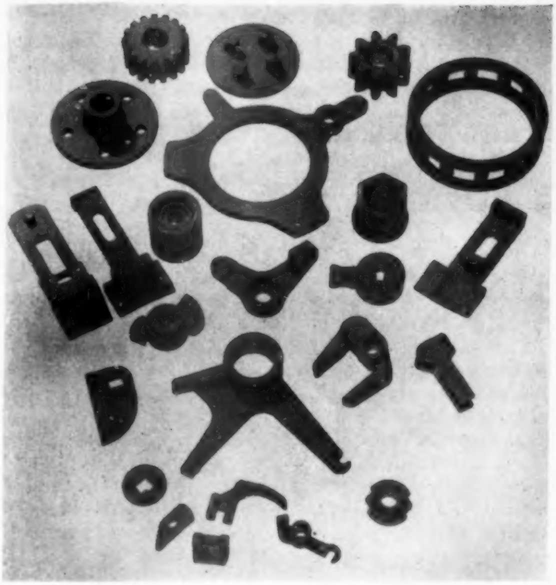
the state of aggregation and flow properties of the powders used. This has been stimulated by advances in the design and types of press used, of which perhaps the most notable examples are among the electrically controlled rapid-acting hydraulic presses being produced in Germany. In many cases, particularly with tungsten carbide powder, where the advantage of using very fine particles is fully recognised, a process known as granulating has been adopted, involving the mixing of the carbide powder with a given quantity of wax, pressing under high pressure and then breaking down into a coarse powder having a useful flow factor. It has been found necessary also to increase the accuracy of the quantity fed into the die at each pressing, and the use of automatic weighing machines coupled directly to the automatic presses is becoming common practice[9]. Hot pressing is increasingly studied and applied to both pressing and coining. Hitherto there has not been any notable publication of actual plant and procedure, but an exception is the very valuable paper published at the 8th Annual Meeting of the American Metal Powder Association[10], at which the subject was frankly discussed in considerable detail and apparatus illustrated and described. In addition to hot pressing, increasing use is being made of hot coining ; an account of practice using conventional heating and pressing plant appeared about the middle of 1951[11]. The practice is now somewhat widely followed where good quality parts are required with good physical properties, high finish and conforming to exacting tolerances.

The matter of dies is of first class importance in considering modern progress in pressing, and a useful review of the subject appeared a few months ago[12] in which the advantages of using carbide dies was tabulated and the question of proper lubrication discussed. Lithium, calcium and zinc stearates, alone or in combination, are advised and the value of external lubrication of sintered parts that are to be coined to high densities is emphasised.

A useful contribution to the general subject of sintering occurs in a discussion on driving bands for shells[13], in which it is explained that many anomalous results obtained with driving bands were traced to insufficient sintering, and that only those bands gave a satisfactory performance which had been so thoroughly sintered as to cause the particle boundaries to disappear. One cannot escape the reflection that many thousands of failures of pressed and sintered parts have also occurred just because of insufficient sintering.

A very interesting item of information included in this particular report on powder metallurgy in National Defence in U.S.A. is that the savings that will result from the adoption of powder metallurgy techniques will probably be in the region of $20,000,000 per annum.

In considering general trends in sintering, especially in Europe, there is no doubt that the use of longer sintering times is growing, also the use of batch sintering as opposed to continuous operation. This is particularly the case with the production of hard metal materials, and with it goes increasing use of vacuum sintering in which high temperature as well as high vacuum is used. Where the lowest possible degree of porosity is essential, it is becoming usual to hot press in a die at sintering temperature. In this procedure, success is largely dependent upon the availability of much improved die materials. Hitherto graphite has been used, but both here and in the U.S.A. the electrical industry manages to get the best material produced. At the moment the most hopeful material for hot pressing dies would appear to be a titanium-carbide-containing composition, with a good resistance to high temperature as well as good behaviour under comparatively high pressures.



Selection of iron parts produced by cold pressing and sintering. Products of Metallwerke Plansee, Reutte-Tyrol. (¼ natural size.)

Bearings

The powder metallurgist will always have a special interest in porous oil-impregnated bearings, but to-day the subject has extended far beyond the simple self-lubricating bearing that provided an outlet for the products of powder metallurgy long ago. The subject holds great interest for the mechanical engineer and has provided inspiration for many important pieces of research, particularly on the theory as well as the practice of lubrication and the mutual reactions of bearing metal surfaces. A very up-to-date account of the properties and applications of self-lubricating bearings was given at the 4th International Mechanical Engineering Congress at Stockholm[14] this year. A notable development in the last year or two is the use of materials for impregnating or lining self lubricating bearings, such materials having lubricating properties. An example is covered by B.P.657,080[15] in which claims are made for the use of the plastic polytetrafluorethylene. This material, in a 5 : 1 mixture by volume of silver powder and the powdered plastic, is pressed on to a silver-plated steel or bronze backing and sintered in a cracked ammonia atmosphere at 360° C. for approximately an hour. Alternatively, the plastic powder may be pressed into small and closely spaced pits formed on the bearing metal surface. In B.P.657,085 the same plastic is incorporated in steel-backed sintered lead-bronze bearings by impressing the powder or sheet into the bearing surface and heating at 350° to 400° C. under a pressure of 1 ton/sq. in. until the plastic fills the pores to a depth of at least 0·03 in. to 0·04 in. Nylon powder and phenolformaldehyde have also been used and their

tendency to overheat can be avoided by mixing a certain amount of sodium carbonate or benzoate when water is the lubricant, or lithium powder or calcium oxide powder when the lubricant is a mineral oil. In this latter case, the mixture and the lubricant combine to form a soap. This procedure is covered by B.P.677,130. Yet another patent, namely B.P.677,240, covers the use of molybdenum disulphide which is worked into steel-backed bearings (sintered) by dusting and squeeze-compacting.

High Temperature Resistant Alloys

Here we reach a field in which active research and development have long been taking place, so that it is almost impossible in a small space to convey a clear idea of the problems that are solved, in process of solution, or awaiting investigation. Great things are expected of powder metallurgy here and the promise is being redeemed. Powder metallurgy has certain advantages, not the least of which is that it can use materials which are not bounded by the limitations of the phase rule. Put very crudely, this means that in powder metallurgy we can use materials that could never exist side by side in a melt. Hence we have the opportunity of using and testing a tremendous range of materials, the influence of which may well provide control over properties hitherto uncontrollable. A group of examples could be taken from the use of oxides and similar compounds as additives controlling, or at least influencing, grain growth, whereby enhanced strength and improved creep properties are obtained, superior to those produced by casting or forging. Another example is the control of the distribution of the double carbide of iron and tungsten in high speed steels. Here the double carbide can be introduced after preparation by any suitable method in powder form and mixed with iron powder, pressed and sintered to give a high-speed steel in which there is no segregation and no formation of carbide-enriched areas, nor of carbide improverished areas such as arise in ordinary metallurgical operations[17]. There is also a huge field in which carbides, borides, oxides and other refractory materials may, and almost certainly will, play most important parts. That any comprehensive survey of so large a subject is impossible may well be obvious if reference be made to the Report on the Symposium on High-Temperature Steels and Alloys[16] published by the Iron and Steel Institute in July of this year. An excellent general review of recent research, particularly Continental, in the field of borides, carbides, nitrides and silicides with 108 references to recent literature appeared in April this year[18].

At present the outstanding problem in high temperature resistant alloys would appear to be the production of turbine blades capable of standing the temperatures that operators are looking forward to in the near future. So great is the volume of work being done, and so numerous the papers describing it, that some suggestions of the trend of recent investigation is all that can be attempted. In the matter of producing the actual turbine blades, an interesting development is the process of shock-compacting, in which the metal powders are compacted in a rubber bag by hydraulic pressure[19] whereby a uniform pressure is obtained over the whole surface; in practice, pressures of the order of 50,000 lb./sq. in. can be applied. A modification of this procedure is the subject of a German patent application[20]. In this case, spherical pressings are made and the method is used in the preparation of titanium and zirconium metals from sponge.

Two important directions of active investigation are the use of highly refractory metals, or mixtures of metals and ceramic powders, to produce the actual blades, or the use of high melting point, but easily oxidised or corroded metals having a protective coating of ceramal type. An interesting example of the first type is covered by an Austrian Patent[21]. It consists of a skeleton of titanium carbide impregnated with an alloy of the Nimonic or Vitallium type and has been used in the production of gas turbine blades, jet nozzles and an annealing container. An example of protective coat comprises a blade consisting of 80% titanium carbide and 20% cobalt upon which a ceramal coat of 80% by weight of chromium and 20% frit is fired at 1,200° C. for 10 minutes in a hydrogen atmosphere. This is claimed to permit of operating the turbine blade so produced at 980° C.[22].

The variety of coatings proposed or actually tested is extraordinary, and some of the claims made for them appear almost too good to be true. It is suggested, for example, that molybdenum metal can be coated or clad as protection against oxidation at high temperatures; alloys are said to be superior to the pure metal in this respect. Using siliconised coatings, it is claimed that the metal can be heated to 1,850° C. for extended periods. If nickel or Inconel cladding is used and suitable protection given to edges, wrought forms can be heated in air to 980° to 1,090° C. Another process[23] consists in giving a highly porous coating which acts as a thermal barrier layer, porosity being produced by using naphthalene as a volatile agent; the coated blades are sintered for from two to three hours at 1,750° C. In contrast to this is a means of producing firmly adherent non-porous coatings on molybdenum, etc.[24]. Molybdenum gas turbine blades are dipped into a molten aluminium-silicon alloy with 36% silicon, at 1,100° C. for 30 seconds to give a 0·012 in. thick coating. This is then converted into sillimanite by heating in air at 800° to 1,000° C. The blades are then coated with a paste of 95% silica, 5% alumina in carbon tetrachloride. This evaporates when heated, and when the temperature reaches 1,600° C. the oxide mixture fuses to a glaze. This process is very close to the patent held by the Fulmer Research Institute[25] which covers molybdenum coated with alumina either by slip-casting or by compacting with a damp mix and firing in an oxygen-free atmosphere. Two useful general surveys of high temperature materials produced by powder metallurgy were published by W. D. Jones[26] and by P. Schwarzkopf[27]. In both cases a careful discussion of the properties required in these materials is followed by a review of work actually done.

Magnetic Materials

One of the most interesting pieces of information regarding metal powder materials for permanent magnets was contained in a brief note in the *Journal of the Franklin Institute* for August[28], to the effect that the Institute Laboratories are to undertake a comprehensive Government-sponsored research into permanent magnet materials produced from powder. Certain theoretical predictions as to relationships between magnetic properties, particle size and other physical properties require checking, and the work is also expected to lead to the discovery of materials having superior

continued on page 298

Recent Developments in Cast Iron Metallurgy

By Arthur B. Everest, Ph.D., F.I.M.*

Research and development work in cast iron metallurgy during the last two years has been directed mainly to various factors influencing graphite formation, and particularly to the production and application of castings with graphite in the spheroidal form. Developments in melting and moulding practice in the foundry have had an important bearing on the improvement of iron castings generally.

PROGRESS has been at a high level in the period since the last review of cast iron metallurgy by Dr. J. E. Hurst, in the November, 1950, issue of *Metallurgia*. In this period two outstanding International Foundry Conferences have been held, in Belgium and the U.S.A., and high attendances have been recorded at National Foundry Conferences both at home and abroad. Progress in the metallurgy of cast iron formed an important part of all of these. In addition, the growing appreciation of the importance of cast iron as an engineering material has also been recognised by the inclusion of papers and sessions on cast iron at various engineering conferences, such as, for example, the International Internal Combustion Engine Conference in Paris in 1951, and the 4th Mechanical Engineering Congress in Sweden this year. It is perhaps particularly significant that the first session at the latter conference was devoted entirely to cast iron.

The output of technical literature in connection with cast iron metallurgy and foundry practice is still at flood level. Papers given during the period have covered all aspects of the technology, foundry practice and engineering application of cast iron, with special reference to spheroidal graphite cast iron. The development of this new material has also stimulated research workers to more extensive studies of the mechanism of solidification of cast iron and of graphite formation. Melting furnace developments include especially the use of basic refractories and hot blast in cupolas, whilst, on the founding side, the shell moulding or "C process," and the use of synthetic resins as core binders are having an important effect on the quality of castings made available to engineers.

During the period several important contributions to cast iron metallurgy have been made by the British Cast Iron Research Association, and of particular significance are the various conferences organised by the Association for the dissemination and exchange of technical information.

Activity in the Institute of British Foundrymen Technical Council and its sub-committees has also continued at a high level. Special reference must be made to the excellent film on the Flow of Metals which was released at the Institute's Conference in 1952. Other aspects of cast iron metallurgy which have been receiving continued study include internal stress in castings, heat treatment, mechanism of solidification, and the influence of mould materials on the structure and properties of castings. The importance of the work carried out by the Technical Council on the reclamation of iron castings was recognised by a review of this subject at the International Foundry Congress in the U.S.A., when the work carried out in this country was reported.

There is little fresh information to add to that given by Dr. Hurst on the general subject of alloy cast irons. All the grades referred to by him under the heading of high duty irons—martensitic, acicular, austenitic and high-chromium irons—have been in continuous production, and have fulfilled the needs of engineers for many special conditions. During the period under review, however, restrictions on the use of alloys, particularly nickel and molybdenum, have been in force, so that both at home and abroad the production of the alloy irons has been restricted to essential purposes.

Spheroidal Graphite Cast Iron

Pride of place in any review of cast iron metallurgy must at this time be given to spheroidal graphite cast iron (S.G. iron, and sometimes also called nodular cast iron, or ductile iron). Since the announcement of the cerium process in 1948, followed by that of the magnesium process, progress in this field has been so rapid that it is difficult even for a specialist to keep up-to-date. The number of technical papers on various aspects of this development now runs into many hundreds, and some idea of the attention being given to it can be gained from the fact that at the International Foundry Congress in 1952, nine of the papers presented were on this subject. This number must be at least doubled to include the more important papers presented at other Congresses in the current year alone. Experience during the last few years has shown that from the practical production point of view, the magnesium process is both cheaper and more widely applicable than the cerium process. Up to the present time nearly 200 firms have been licensed throughout the world for the production of spheroidal graphite cast iron under patents and patent applications, covering the magnesium process, in the names of The International Nickel and Mond Nickel Companies. Production in the foundries of these licensed firms is, of course, only now building up to substantial proportions. Acceptance of the new material by the engineering industry is inclined to be slow, since, for many applications, protracted service trials have to be undertaken before existing specifications are changed. Abundant evidence is, however, available that for many applications S.G. iron is now accepted, and production is proceeding on an ever increasing scale.

* The Mond Nickel Company Ltd., London.

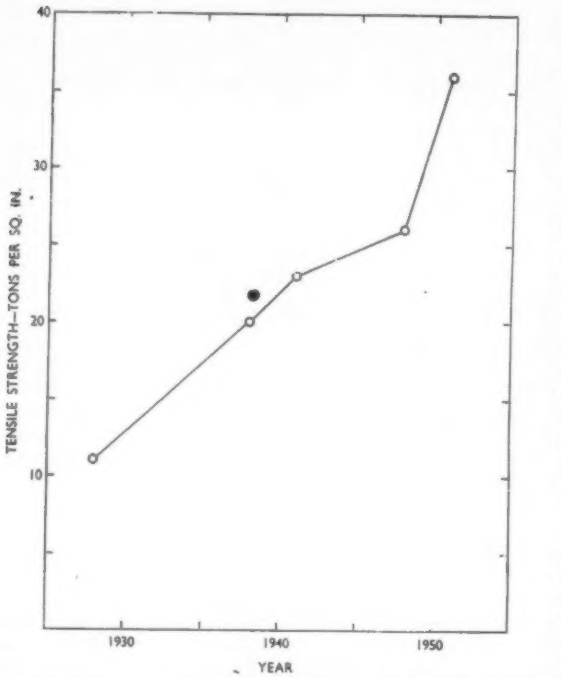


Fig. 1.—Diagram showing improvement in the properties of cast iron over the last twenty-five years, based on the minimum tensile strength of the highest grade of cast iron covered by specification. The figures for 1928, 1938, 1941 and 1948 are based on B.S. 321, 786 and 1452 respectively. The figure for S.G. iron in 1951 is based on the U.S. specification. A.S.T.M. Standard A339-51T.

Fig. 1 shows, in a striking manner, the progress made in cast iron metallurgy in the last 25 years, and indicates the contribution made by S.G. iron. The figures plotted in Fig. 1 show the minimum strength specified for the highest grade in general cast iron specifications. It should be noted that in 1928, the highest strength which it was felt could safely be quoted as a minimum for iron castings was no more than 11 tons/sq. in. This figure was rapidly raised as progress was made with the alloying, inoculation and improved melting of cast iron in the foundry. B.S.1452 in 1948 covered cast irons made in accordance with all the latest developments in cast iron metallurgy, the highest grade corresponding to the acicular type of nickel-molybdenum cast iron. The figure of 36 tons/sq. in. minimum tensile strength specified for S.G. iron in 1951 is taken from a U.S. tentative specification. Corresponding tentative specifications are in use in several other countries, and this figure fairly represents the minimum strength which is to be expected from normal spheroidal graphite iron in the as-cast state, i.e., in the pearlitic condition. At this point it might be worth mentioning that figures well over 60 tons/sq. in. have been quoted for alloyed and heat treated S.G. iron: a truly astonishing figure when considered in relation to cast iron as known only 25 years ago.

In presenting strength data in Fig. 1, it should be emphasised that these figures are put forward merely as a guide to the improvement in mechanical properties. The advantages from the engineers' point of view of the latest types of cast iron reside mainly in properties other than tensile strength, and in this connection particular reference must be made to the increased toughness of the new cast irons, this amounting to some four times the shock resistance when S.G. iron is used in the as-cast, pearlitic condition, and 12 times for S.G. iron in the annealed ferritic state. From the engineers' point of view, the definite and high yield strength of S.G. iron is particularly important, as well as its high elastic modulus and fatigue strength.

In conjunction with these properties, it must be mentioned that outstanding advantages of the new cast iron lie in its ready castability and good machining characteristics, in both of which respects it is far superior to cast steel. A significant point in this connection is that S.G. iron has many of the good casting properties of ordinary cast iron, and does not suffer from any deterioration of properties with increase in carbon content, so that the advantage of high carbon from the castability point of view can be used to the full. For this reason S.G. iron is being used to an increasing extent for the production of complex castings, difficult to produce in cast steel.

Research Investigations

The attention of research workers in ferrous metallurgy throughout the world has been concentrated in the last few years on some of the problems suggested by the development of S.G. iron. Although many theories of a more or less simple nature have been expounded to show why, after many hundreds of years of cast iron production, it has suddenly been found possible to cast grey iron with spheroidal graphite, none of the theories so far put forward really offers a satisfactory explanation to the metallurgist. It is felt that once the reasons for the production of spheroidal graphite by magnesium and cerium treatment are fully understood, a vast new field of research and development will be opened up for exploitation. At the moment, however, we are in the position that magnesium and cerium alone offer a practical method of producing the new iron. Other elements have been shown to be capable of producing spheroidal graphite, but only under conditions which are incapable of reproduction in a practical foundry process. Meanwhile, the whole question of graphite formation in cast iron is being studied both in conventional and malleable cast irons. It is probable that, in the next few years, our knowledge of graphite formation in cast iron will be greatly extended, with beneficial results for the foundry industry as a whole.

Practical foundry processes for the production of S.G. iron have been developed, and are successfully being applied to the production of engineering castings of all sizes and types. A great deal of attention has been given to the study of alternative and cheaper methods of introducing magnesium into cast iron. Nickel is the best carrier for magnesium yet found, and inferior results are obtained when other carriers, such as copper or ferro-silicon, are used. However, research on alternative methods of introduction continues, and considerable importance is attached to research work during the year on methods of making the addition, in the form of metallic magnesium, direct to the iron in the ladle. Considerable success has been achieved using special methods for plunging metallic magnesium, but here the reader must be warned not to carry out any experiments for himself without proper guidance, since magnesium has a boiling point well below the temperature of molten iron, and without proper precautions there is serious danger of explosion.

Detailed information has now been published regarding the effects of interfering elements which were responsible for the fact that in earlier development work some types of pig iron were found to be incompletely responsive to magnesium treatment. The problem raised by these interfering elements has now been studied intensively, and particularly by the B.C.I.R.A. and The Mond Nickel Company. An interesting paper on this subject was presented by Morrogh at the 1952 International Foundry Congress. In this it was shown that the deleterious effects of these elements can be overcome by the introduction of a small proportion of Mischmetall during the magnesium treatment. In the discussion of this paper a contribution was made by the Mond Nickel Company, demonstrating that the proportion of Mischmetall necessary was extremely small. The practical outcome of this work is that to-day fully spheroidal graphite structures can be produced in iron castings almost regardless of the purity of the raw materials used. It must be emphasised, however, that for the best results a careful control over composition is necessary. For example, it is now well-established that higher silicon will give greater strength and hardness, but will lower the ductility and toughness of the castings, so that silicon must be adjusted in accordance with the properties required in the final castings. Manganese, likewise, must be controlled to low levels for maximum ductility. The general trend of development based on our fuller understanding of S.G. iron is towards making the process ever more reliable, whilst at the same time lowering production cost.

Other developments, such as the use of the basic cupola, and even of the "C process," have an important bearing on the development of spheroidal graphite cast iron. Reference is made to these below.

Applications of Spheroidal Graphite Cast Iron

The whole question of the engineering application of the new cast iron has been considered extensively in papers presented at conferences throughout the world. Fields of application for S.G. iron have been proposed on the basis of the properties which the new materials offer. Some of the applications currently in use are dictated by economic conditions, such as, for example, the difficulty in many countries of obtaining quick delivery of steel castings, but a review of experience up-to-date leads the author to indicate the following as the more important directions in which spheroidal graphite cast iron is being applied in industry :—

(1) Iron castings, where improved toughness is required whilst retaining the other good properties of cast iron, such as its castability, machinability, corrosion resistance, etc. A particular field of application in this connection is for marine castings where increased toughness is required to withstand the shocks and stresses normal to marine service. In this field cast iron has about five times the corrosion resistance of steel, and this is of the greatest significance for bedplates, machine frames, brackets, valves, fittings, etc.

(2) For the replacement of steel castings where, due to the better castability of S.G. iron, forms which previously gave great difficulty as steel castings can now successfully be made : for example, compressor heads and complicated valve parts for which ordinary cast iron was not strong enough and which defied the best practice of the steel founder, due to the form being unsuitable for production as steel castings. In

Courtesy of Fonderia Pensotti, Italy.

Fig. 2.—S.G. iron scaffolding clamp, saving 15 per cent. weight over steel or malleable cast iron.

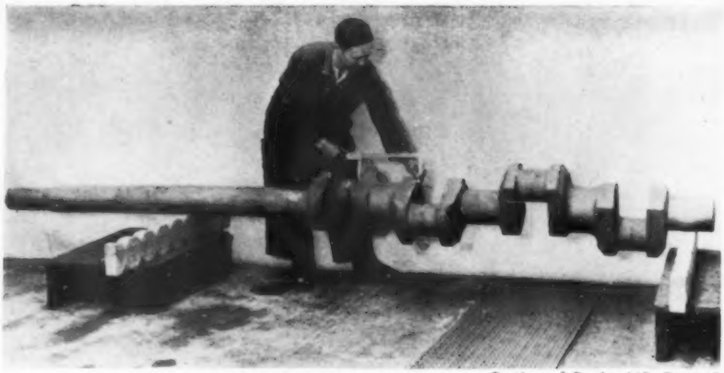
this category must also be included a number of parts where steel had previously to be used, as cast iron was deficient in the necessary strength, but where the corrosion-resistance, good bearing properties and other characteristics of cast iron are desirable. A good example in this connection is for crankshafts, for which S.G. iron is now being adopted for some types of Diesel engines and compressors.

(3) It will be appreciated by those who have studied the literature that S.G. iron has properties closely akin to those of malleable cast iron. In fact, where desired, S.G. iron can replace malleable for many applications. Of greater significance, however, is the fact that due to its nature and characteristics, S.G. iron is not limited to any section thickness. In fact, castings, such as anvil blocks up to 36 in. in section and weighing 50 tons, have successfully been made in S.G. iron and show high strength and ductility even in such heavy sections. Consequently, S.G. iron can be regarded as extending in a practical way the field of application of malleable up to any section.

Whilst the above three fields are, in the author's opinion, currently the most important in the development of S.G. iron, there are innumerable other directions in which development is taking place, such, for example, as in offering reduced section thickness with lighter weight, providing particular properties for specialised types of casting, and for replacing other materials where the particular properties of cast iron are desired.

Types of S.G. Iron

It has been indicated in the literature that spheroidal graphite structures can be produced in all types of grey cast iron normally having graphite in the flake form. What has been said above applies to the normal type of S.G. iron used either as-cast or after a ferritising anneal. For certain purposes, S.G. iron is being used to an increasing extent in the heat-treated condition to give higher strength, toughness and wear resistance for crankshafts, camshafts, gears and other items. The highest strength figures ever recorded for cast iron have



Courtesy of Dania A/S, Denmark.

Fig. 3.—Four-throw crankshaft, over 13 ft. long in S.G. iron.

been obtained with a nickel-molybdenum S.G. iron with an acicular structure, but perhaps of more practical importance are the austenitic S.G. irons.

One of the deficiencies of the austenitic irons, including Nomag, Ni-Resist and Nicrosilal, has been their relatively low mechanical strength, tensile figures of 12 to 14 tons/sq. in. being typical. As in the case of other types of cast iron, the act of rendering the graphite spheroidal not only more than doubles the tensile strength, but, as indicated above, leads to a nearer approach to the steel-like properties of the matrix ,with much greater toughness, a definite yield point, higher modulus and other desirable characteristics. An example of what is being done with austenitic S.G. iron is to be seen in the new metal—Nodumag*—the spheroidal graphite version of Nomag*, for which a tensile strength of 25–28 tons/sq. in., with a yield point of 13–17 tons/sq. in., and an elongation of 10–20% is now claimed. Similiar figures are available in Ni-Resist, coupled with the good corrosion resistance, characteristic of that high alloy cast iron. Particular importance is likely to be attached to these S.G. austenitic irons, since it is clear that they can have many useful applications in chemical plant, oil refineries and the like to replace stainless steel or other materials with inferior casting properties.

Another direction in which S.G. iron is showing outstanding importance is for rolls for metal working. Sufficient attention has now been given to this application for it to be said that a new field is opening up whereby certain types of rolls can be made having the mechanical strength of steel combined with the good wear resistance and frictional properties of cast iron. Some continental firms have already extensively exploited this field, and many hundreds of S.G. iron rolls have fulfilled the highest expectations in service.

All normal casting methods can be applied to S.G. iron, and another application of considerable industrial importance is that of centrifugally cast pipe. Already many hundreds of tons of such pipe have been made and have demonstrated the possibility of replacing cast iron where greater strength and toughness, or reduction in section are desired.

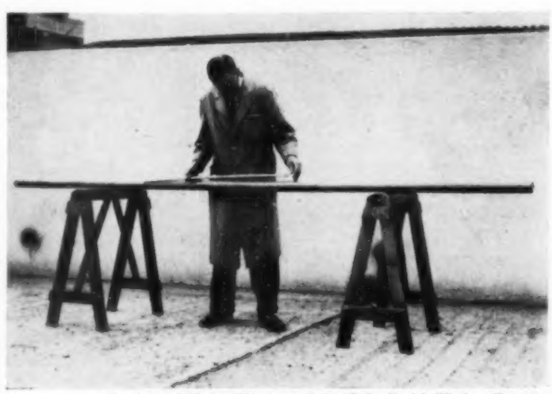
Research has been carried out in the last two years on the hot working properties of spheroidal graphite iron. As might be expected from its structure, the possibilities of hot working the new iron are substantially greater than those for ordinary flake graphite iron. Fig. 4 accompanying this article is of particular interest, in that it shows a 14 ft. long, 2 in. bore pipe which was successfully extruded in S. G. iron under ordinary commercial conditions. S.G. iron has also been found to possess useful properties as a material for hot stamping. These developments are perhaps mainly of academic interest at this time, but there is no doubt that the general adaptability of S.G. iron opens up many new fields for exploitation in cast iron metallurgy.

* Registered Trade Mark—Ferranti Limited.

Alloy Cast Irons

In spite of what has been said above concerning the development of spheroidal graphite cast iron, there are many directions in which the flake form of graphite is desired to confer special properties in the castings. It must not be overlooked that modification of the graphite to the spheroidal form reduces the good property of cast iron in damping vibrations. Thus for many parts, such as machine tool beds and frames and certain engine components, it would be a mistake to consider changing over to the higher strength iron without due regard being paid to this factor. Again, the presence of graphite in the conventional flake form has certain advantages in lubrication in sliding parts, such as cylinder liners, whilst the graphitic structure of cast iron is believed to be advantageous in parts such as brake drums. Flake graphite in items such as ingot moulds also probably contributes to their resistance to thermal shock. In all these directions, S.G. iron may have advantages on account of its increased strength, the possibility of reducing section thickness and the like, but it is believed that there will always be at least some applications for which high-duty flake graphite iron will be required.

As indicated above, restrictions in the availability of strategic elements have prevented to some extent the exploitation of alloy irons into new fields, except where urgently required in the national interest, but some progress has been made in the use, for example, of the nickel-molybdenum irons of the flake graphite type for brake drums and other parts where an open structure iron is desired. Again, the flake graphite acicular type of iron continues to be used for cast crankshafts.



Courtesy of Henry Wiggin and Co. Ltd., Zenith Works, Glasgow.

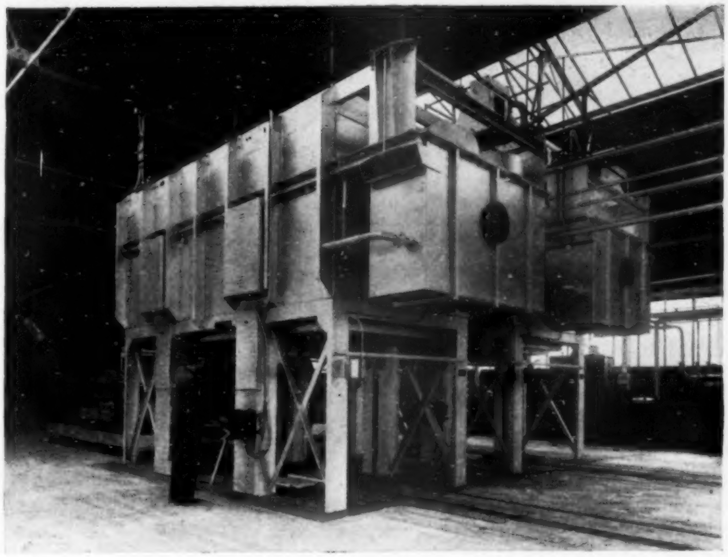
Fig. 4.—Extruded S.G. iron pipe, 14 ft. long, 2 in. internal diameter.

In the field of alloy cast irons, special reference must also be made to Ni-Hard—the alloy white iron which has proved outstanding and is now widely used in giving maximum resistance to wear in rolls for the metallurgical industry, and for crushing and grinding equipment as used in the mining and other industries.

Melting Furnace Developments

In the practical development of S.G. as well as other types of modern cast iron, continuous interest has been shown in methods of cheapening production by the use of lower cost base materials, and in particular the increased use of steel scrap. Considerable attention was given at the International Foundry Congress to reports on the development of the basic cupola, especially when associated with hot blast. From the point of view of foundry metallurgy, this development is of considerable importance in the possibility which it provides of the use of cheaper raw materials to provide an iron of suitable composition, and notably in having a high carbon, when desired, and low sulphur content. The idea of the basic cupola was initiated in this country, but like some other developments, its practical exploitation has taken place mainly in America, where already a large number of installations are in operation. Some of these installations are highly complex, such as, for example, that at Lynchburg Foundry, where the basic cupola, equipped with hot blast, water cooling and other refinements, has a control room worthy of a small power station. On the simpler scale, however, the basic cupola undoubtedly offers interesting advantages for the production of high-duty iron of all types.

In countries where electric power is cheap and plentiful, developments continue in the application of electric melting to cast iron, notably in connection with large low frequency furnaces capable of melting scrap and even cast iron borings on a large scale.



Courtesy of Birlec Ltd., Birmingham, and Bullers Ltd., Tipton.

Fig. 5.—A modern electric furnace for the gas annealing of malleable cast iron.

The "C Process"

Though not strictly related to the metallurgy of cast iron as such, passing reference must be made to the shell moulding or "C process," in view of its great importance to the foundry industry and to engineers. Briefly, the advantages of the "C process" lie in the facilities offered for the production of precision castings under ordinary foundry conditions. Again, it can be envisaged that it may not be necessary for an individual foundry to instal the necessary plant and equipment for operating the shell moulding process on its own premises, and we may in the future have firms established to provide shell moulds to foundries when a small number of precision castings are required. Already there is evidence from the U.S. that the development of spheroidal graphite cast iron is being married to the "C process" for the provision of high quality precision castings for the most exacting engineering applications.

Heat Treatment

Fig. 5 shows a modern gas annealing furnace as applied to the malleable industry. The development of gas annealing goes back some years, but it is now proving of the greatest importance, not only in economically heat treating white iron castings for the malleable process, but on account of the improved quality of product and the general improvement in the cleanliness and appearance of the malleable shops. Considerable importance is likely to be attached, in the future, to annealing furnaces with gas atmospheres, since associated with the development of new types of cast iron is also the increased use of heat treatment of iron castings to develop particular properties. In this connection, gas atmospheres are important, and a development of the future is likely to be the electric annealing furnace with controlled atmosphere to minimise scaling.

Conclusion

Anyone conversant with foundry literature will appreciate fully the intense level of research and development at present being applied to cast iron and its foundry practice. In a brief article of this type it has been possible only to sketch some of the more important developments in this field. In closing, however, reference must be made to the increased awareness in the industry of the necessity for exchange of information. Reference has been made above to the increasing popularity of congresses where problems and new information are discussed in public. In the period under review, considerable activity has been seen under the heading of productivity teams, whereby foundrymen and others have been given an opportunity of studying methods in the United States. The exchange of experience of this type will undoubtedly lead to improved results in all aspects of foundry practice. Likewise, the increased popularity of International Conferences and the work of International Committees best contribute to the desire of foundrymen throughout the world to get together for the mutual improvement of their practice and technology.

How Powder Metallurgy is Progressing

Continued from page 292.

properties to those now in use. A most useful survey along somewhat similar lines has been published recently on the production and application of magnetic powders by G. R. Polgreen[29]. There was also a valuable paper given by W. Hotop before the 4th International Mechanical Engineering Congress at Stockholm[30] in which a useful discussion is presented of technical and economic factors aiding the development of pressed and sintered magnets by which they have now become an important and a valuable adjunct to cast permanent magnets. The number and volume of papers, patents and communications on magnets and magnetic materials grows constantly. An entirely new magnetic material has been described recently[31]; it is composed of bismuth and manganese and has been developed by the Naval Ordnance Laboratory, White Oak, Ma., U.S.A. The new material has a flux density exceeded only by platinum-cobalt alloy. Magnets can be made by powder metallurgy techniques, thus ensuring close tolerance of complicated shapes without machining. Production involves heating powdered manganese and bismuth at 700° C. and hot pressing the resulting bismuthide at 300° C. in a strong magnetic field. The material exhibits a coercive force of 3,000 Oersteds, greater than any known permanent magnet material.

Of necessity, this short review of progress in powder metallurgy during the last couple of years is very incomplete and very patchy. It may, however, convey some idea of the very real progress being made in all the industrial nations of the world. In a review of this kind, one naturally selects the high-lights, but they are in sufficient number and of adequate brilliance to illuminate the whole picture. It is particularly satisfactory to note that this country manages to maintain a sound position in respect of both research and industrial development, and continues to contribute in no mean fashion to the technological and scientific progress of powder metallurgy.

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Review of Recent Developments in Electroplating

Continued from page 288.

differences were found in the rate of removal of the stearic acid and the amount of residual contamination left. With each cleaning solution used, an equilibrium value tended to be reached, after which the degree of contamination did not decrease further with increased cleaning time. With a cleaning time of 2 minutes at 90° C., the concentration of the alkaline solutions tested had no appreciable effect on the removal of stearic acid over the range of 0·1% to 1·0% Na_2O and an increase in concentration above 1% actually resulted in a very rapid increase in residual contamination on the test piece.

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Safety and Factory Efficiency Exhibition

THE Birmingham Industrial Safety Group announce their intention of holding a Coronation Year Exhibition in the Bingley Hall, Birmingham, from 19th to 26th June, 1953, inclusive. This Exhibition will follow the pattern of the highly successful Exhibition which was organised in Festival Year and which attracted more than 10,000 visitors in the four days during which it was open. The scope of the Exhibition is being extended to cover all aspects of Safety, Efficiency and Health in Industry.

On this occasion the whole of the Bingley Hall has been booked, and to coincide with the Exhibition a week-end Conference is being organised with emphasis on the theme of training for Safety and Health. This Conference, which will draw representatives from all parts of the United Kingdom will open on Friday, 19th June. Papers will be given by leading authorities on works training methods with particular emphasis on accident prevention.

The Birmingham Group draws its members from practically every large industrial concern in the Midlands: in addition to 170 large undertakings, more than 290 smaller firms are connected with the organisation through associate membership.

An attractive brochure dealing with the Exhibition has been published and copies of this with any further information may be obtained from Mr. A. G. Cogswell, S.A.F.E. Exhibition Secretary, Fort Dunlop, Birmingham, 24.

The Properties of Some Binary Aluminium Alloys at Elevated Temperatures

By J. V. Lyons*, Ph.D., and W. I. Pumphrey†, M.Sc., Ph.D.

In many metal working operations, and particularly in the fabrication of complex structures by welding, aluminium alloys may be subjected to stress whilst at a temperature which is approaching their melting points. In an attempt to determine the factors which affect the mechanical properties of aluminium alloys at temperatures both above and below the solidus, an examination has been made of the high temperature tensile properties of alloys in a number of the more common binary alloy systems. The results obtained in this examination were reported in the November issue of this Journal and the implication of these results is considered below.

5. Discussion

The strength/temperature curves of all the alloys examined during the course of the present investigation were of the same general form. The theoretical implications of the general form of the curve showing the variation of the strength of an aluminium alloy with temperature have already been considered by Singer and Cottrell[1] and by Pumphrey and Jennings[6]. The tendency of aluminium alloys to crack at temperatures above the solidus is dependent upon the mechanical properties of the alloys at such temperatures, and in the light of the results obtained in this investigation it is of interest to consider the behaviour of alloys in the systems aluminium-copper, aluminium-iron, aluminium-manganese and aluminium-zinc in tests designed to estimate the tendency of such alloys to crack during casting and welding.

Pumphrey and Jennings[7] have shown that cracking in aluminium alloys at temperatures above the solidus occurs in the brittle temperature range between the effective solidus temperature and the temperature at which a coherent dendrite structure first forms in the alloy during cooling from the liquid state. Such cracking is a result of restraint of the free contraction of the solidifying metal. The inherent tendency of an alloy to crack at temperatures above the solidus is directly proportional to the extent of the brittle range, and the extent of this range with any alloy may be determined from tensile tests carried out on suitable specimens of the alloy heated to the temperature of testing from the solid state. In such conditions the brittle range is that range of temperature between the temperature at which liquid first appears between the crystals of the alloy (as manifested by the first appearance of brittle fractures in the tensile test) and the temperature at which the alloy loses all coherence or strength. The brittle range of every alloy examined during the present investigation has been determined.

In Fig. 16 the limits of the brittle temperature range for the various aluminium-copper alloys examined are indicated on a structural diagram. This diagram also indicates the effect of the degree of approach to structural equilibrium on the extent of the brittle temperature range in each of the aluminium-copper alloys tested.

The lowest curve in Fig. 16 is a hypothetical curve relating to a cast alloy heated at an infinitely rapid

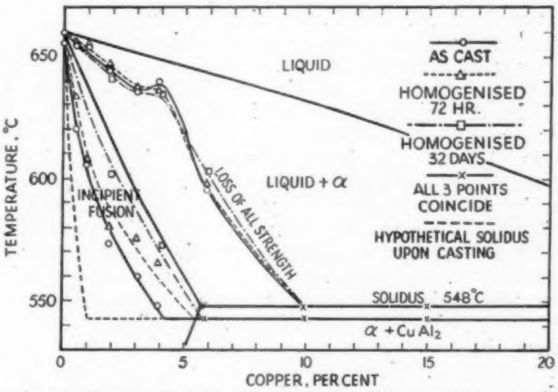

Fig. 16.—Temperature of incipient fusion and loss of all strength superimposed on a portion of the aluminium-copper equilibrium diagram.

rate of heating to the temperature at which the tensile test is carried out. If such a rate of heating were possible, an alloy would retain its cast structure at the temperature of testing; in this event, as has been shown by microscopic examination of the cast bars of the aluminium-copper alloys, incipient fusion would occur at the eutectic temperature in an alloy containing as little as 1% of copper.

This condition is simulated in Fig. 16 by projecting a temperature horizontal at 543° C. to meet the vertical composition line at 1·0% copper and completing the effective solidus line from this meeting point up to the melting point of pure aluminium.

If it is assumed that the temperature at which all coherence is lost is not appreciably different from that found with the cast specimens actually tested, then the brittle temperature range for aluminium-copper alloys under actual casting conditions can be derived and plotted as in Fig. 17. The curves in this figure indicate the tendency to cracking at temperatures above the solidus during casting and welding of the series of aluminium-copper alloys examined. The results obtained by Pumphrey and Lyons in ring-casting and restrained weld tests of aluminium-copper alloys are reproduced in Fig. 18. These investigators also reported the presence of the eutectic phase in an aluminium-copper alloy containing 1% of copper; hence, their results on the tendency to cracking in aluminium-copper alloys should correspond with the hypothetically derived

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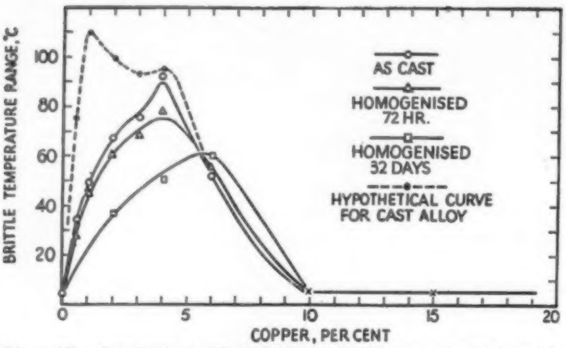


Fig. 17.—Variation of brittle temperature range with copper content of aluminium-copper alloys (derived from curves of Fig. 16).

curve, included in Fig. 17, showing the variation of extent of brittle temperature range with copper content. Comparison of Figs. 17 and 18 confirms that there is close agreement between the positions of the maxima of the curves and between the actual shapes of the curves.

Previous mention has been made of the appreciable amount of inverse segregation found in the cast bars of the aluminium-copper alloy containing 4% of copper. This defect was present in the tensile specimens turned from the cast bars; consequently, when the cast alloys were tested at elevated temperatures, the core of each test-piece behaved as an alloy containing less than 4% of copper, whilst the case behaved as an alloy containing more than 4% of copper. The core of the test-piece thus retained coherence to a higher temperature than the case and because of this the apparent brittle temperature range of the alloy containing 4% of copper—as determined by tensile testing—was greater than that of the alloys containing either 3% or 5% of copper; this is shown by the curves in Fig. 16. An alloy in which inverse segregation occurs during casting will, therefore, have a more extended brittle temperature range than an alloy in the same system of somewhat higher or lower alloy content in which inverse segregation does not occur. This is shown by the form of the upper curve in Fig. 17. From the form of this curve it would be expected that when aluminium-copper alloys are cast and caused to solidify under restraint, more cracking would occur in an alloy containing about 4% of copper

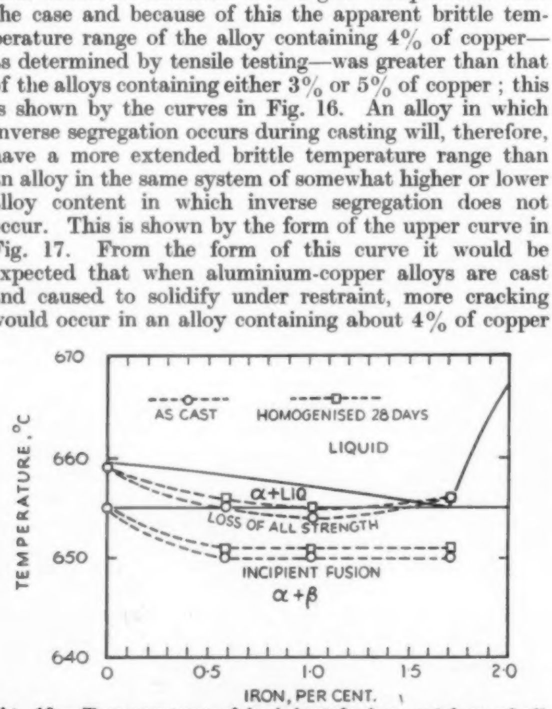
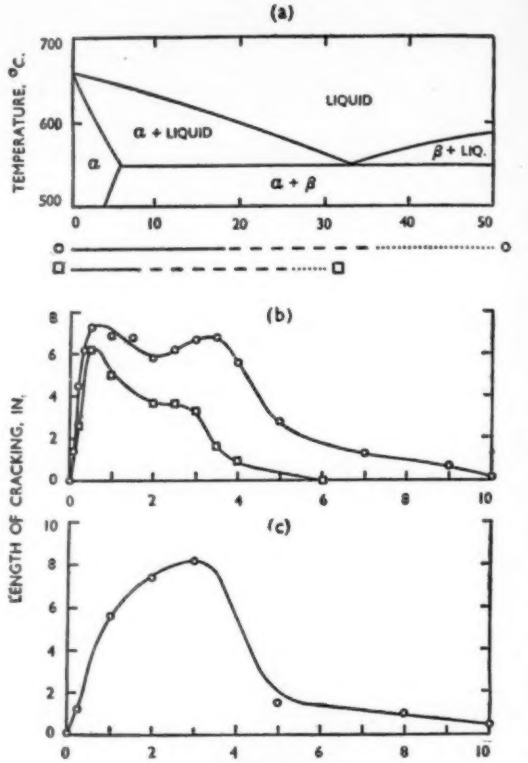


Fig. 19.—Temperature of incipient fusion and loss of all strength superimposed on a portion of the aluminium-iron equilibrium diagram.



Courtesy of the Journal of the Institute of Metals.

Fig. 18.—Cracking of ring castings and restrained welds in the aluminium-copper system (Pumphrey & Lyons). (a) Equilibrium diagram; (b) cracking of ring castings; (c) cracking of restrained welds.

than in alloys containing either 3% or 5% of copper. The experimental results obtained by Pumphrey and Lyons[3] show that this expectation is, in fact, realised, and the form of the curve obtained by these workers showing the variation with copper content of the tendency of aluminium-copper alloys to crack during casting closely corresponds with that of the upper curve in Fig. 17. It thus seems reasonable to assume that the anomalous second maximum on the cracking curve published by Pumphrey and Lyons is ascribable to the occurrence of inverse segregation during solidification in the alloy containing 4% of copper. A recent microscopic examination of the ring test-pieces cast by the present workers in their earlier experiments has confirmed that inverse segregation was, in fact, present in the cast rings of the

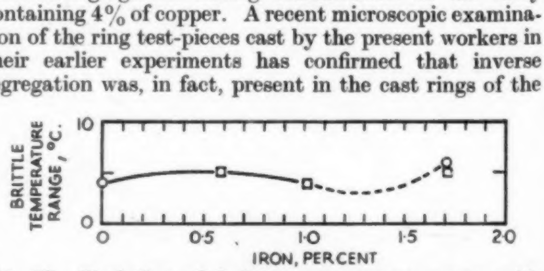


Fig. 20.—Variation of brittle temperature range with iron content of aluminium-iron alloys (derived from Fig. 19).

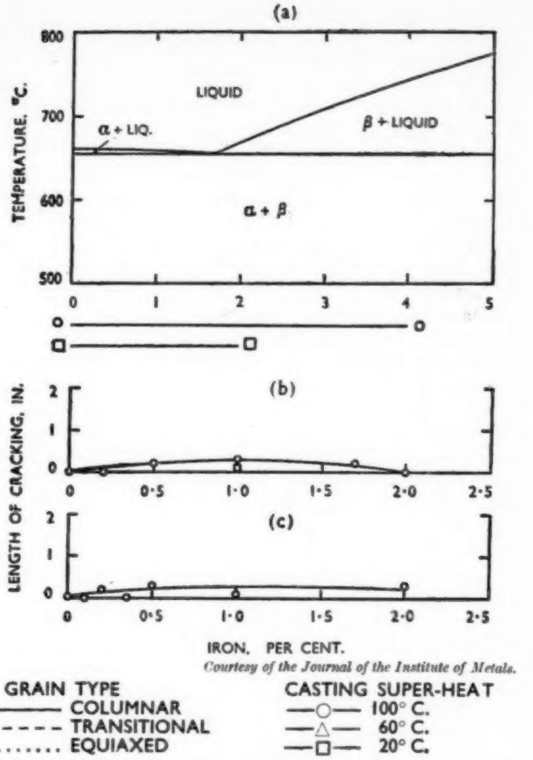


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Fig. 21.—Cracking of ring castings and restrained welds in the aluminium-iron system (Pumphrey & Lyons). (a) Equilibrium diagram; (b) cracking of ring castings; (c) cracking of restrained welds.

aluminium-copper alloy containing 4% of copper but not in the cast rings of the other aluminium-copper alloys.

Inverse segregation is not reduced, to any appreciable degree, by even lengthy homogenising treatments; in fact, some effects of inverse segregation in producing the duplex fracture mentioned previously were still observable in those specimens of the aluminium 4% copper alloy homogenised for 32 days. The effects of inverse segregation were considerably reduced after this period of homogenising, however, as is shown by the smaller brittle temperature range in the 4% copper alloy homogenised for the longer period.

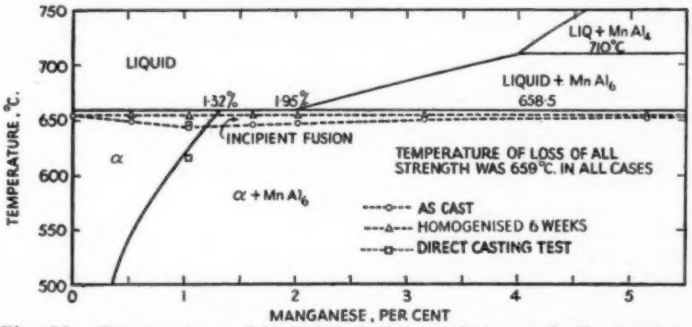

Fig. 22.—Temperature of incipient fusion and loss of all strength superimposed on a portion of the aluminium-manganese equilibrium diagram.

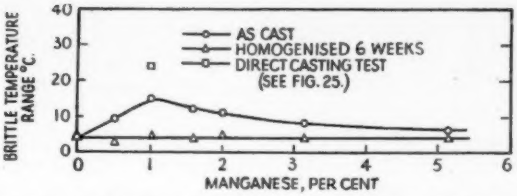

Fig. 23.—Variation of brittle temperature range with manganese content of aluminium-manganese alloys.

The conditions prevailing during a welding operation, in which the sheet is itself in a state very nearly approaching structural equilibrium, and in which the rate of cooling of the deposited weld metal is, perhaps, slower than that of a ring casting, will probably produce structures which are nearer to a state of equilibrium than those resulting from the casting operation. Consequently, it seems that the experimental curves in Fig. 17—relating to alloys reheated from the cast condition and to alloys heated to the testing temperature after homogenising for 72 hours—provide some indication of the extent of the brittle temperature range in aluminium-copper alloys in conditions of welding, and hence enable some estimate to be made of the probable tendency of such alloys to crack during welding. The general form of these curves is in good agreement with that of the curve published previously[3] by the present authors showing the experimentally determined variation with copper content of the tendency of aluminium-copper alloys to crack during welding.

The temperatures at which brittle fractures first occur, and at which all coherence is lost, in tensile test-pieces of aluminium-iron alloys, are plotted on a structural diagram of the aluminium-iron system in Fig. 19. There are, however, insufficient experimental points to permit the construction of accurate curves, and the diagram of the brittle temperature range in Fig. 20 is constructed from the actual critical points determined for each alloy.

It is apparent from Fig. 20 that the brittle temperature range of the three aluminium-iron alloys examined is small—of the order of 5° C.—and, therefore, these alloys should exhibit only a very small tendency to cracking during solidification. This suggestion is in agreement with the results obtained by Pumphrey and Lyons with aluminium-iron alloys tested in the ring casting and the restrained weld test (Fig. 21).

The temperatures at which incipient fusion occurred (that is, the temperatures at which brittle fractures were first observed in the tensile tests) and the temperatures at which all coherence was lost in the aluminium-manganese alloys are shown superimposed on a portion of the equilibrium constitutional diagram for aluminium-manganese alloys in Fig. 22, and the variation with manganese content of the extent of the brittle temperature range of the alloys is shown in Fig. 23. The brittle temperature range is small for all the alloys tested in the cast condition but appears to increase to a maximum in the alloy containing 1% of manganese and then to decrease to a small value at higher manganese contents. This is in agreement with the variation, with com-

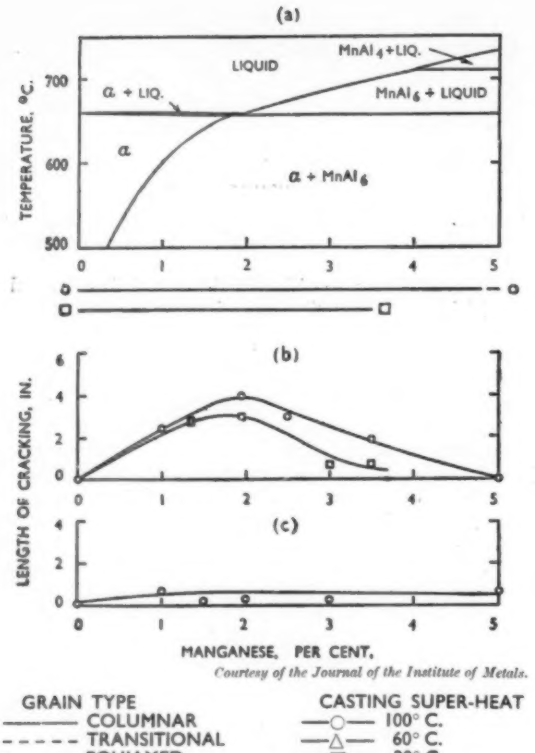

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Fig. 24.—Cracking of ring castings and restrained welds in the aluminium-manganese systems (Pumphrey & Lyons). (a) Equilibrium diagram; (b) cracking of ring castings; (c) cracking of restrained welds.

position, of cracking in ring castings observed in previous work[3].

The brittle temperature range of all the homogenised alloys is small, being approximately 4° C., which is in accordance with the experimental observation that only slight cracking is observed in welding tests carried out on these alloys (Fig. 24). Despite this general agreement with results obtained in welding tests, however, the extent of the brittle temperature range of the alloys appears to be insufficient to account for their observed tendency to crack during casting. Pumphrey and Lyons found that certain of the aluminium-manganese alloys contained particles of $MnAl_6$ in their crystal boundaries when solid, and postulated that the presence of such particles would cause fracture at the crystal boundaries under a sufficiently high stress. They suggested that while the stresses set up in the metal of the castings used in their experiments were sufficient to cause such fracture, the stresses in the weld metal were not, and they argued that the increased amount of cracking in the cast alloys was due to cracking occurring at temperatures below the solidus during the cooling of the cast metal.

As already mentioned, cracking during the initial stages of deformation did occur in the gauge length of test-pieces of certain of the aluminium-manganese alloys tested at temperatures below the solidus. This result appears to confirm the validity of the explanation previously advanced by the present authors to account for the pronounced tendency of aluminium-manganese alloys to crack in the ring-casting test. It is possible, however, that the greater tendency to cracking of the alloys when cast than when welded may be due to an increase in the extent of the brittle temperature range in the cast alloys brought about by the occurrence of undercooling during solidification after casting. In order to determine whether such undercooling does, in fact, occur in aluminium-manganese alloys, tensile tests were carried out on an aluminium-manganese alloy containing 1% of manganese during cooling from the liquid state. The apparatus employed for these tests was that used previously by Pumphrey and Jennings for similar tests on aluminium-silicon alloys[6]. The results obtained in the tests are recorded in Fig. 25 and indicate that the occurrence of undercooling in the alloy containing 1% of manganese caused a considerable increase in the brittle temperature range of this alloy.

It is possible, therefore, that two factors are responsible for the large amount of cracking which occurs in ring castings of aluminium-manganese alloys; the first of these is the increase in the brittle temperature range produced by undercooling during rapid solidification, and the second is the increased brittleness of the alloys at sub-solidus temperatures caused by the presence of hard particles of the $MnAl_6$ phase in the crystal boundaries. It seems probable, however, that the first of these two factors is of greater importance than the second.

It is conceivable that neither of the two factors mentioned above is predominant in the restrained weld test. In this test, cooling of the solidifying metal is probably less rapid than in the ring-casting test so that the brittle temperature range is not increased to any marked extent by undercooling of the solidifying metal. Furthermore, unmelted parent metal forms the greater part of the cooling metal in the restrained weld test and — as shown by the tensile tests on homogenised aluminium-manganese alloys—this wrought material is more ductile and weaker than the cast metal. Thus, any stresses which arise in the weld are probably reduced by elongation of the parent sheet before the stresses reach a sufficient magnitude to cause cracking of the brittle weld metal in which, as in cast metal, particles of $MnAl_6$ at the crystal boundaries promote embrittlement. This consideration possibly explains the smaller amount of cracking observed in restrained welds than in ring castings of aluminium-manganese alloys.

The temperatures at which incipient fusion occurred in test-pieces of the aluminium-zinc alloys are shown

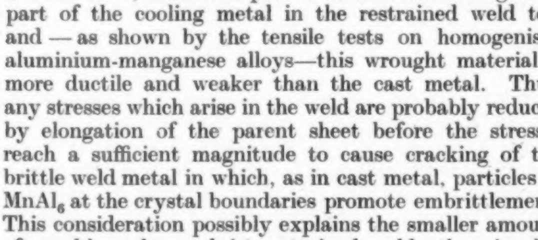

Fig. 25.—Variation of strength with temperature for an aluminium-1% manganese alloy as determined by the direct casting test.

superimposed on the equilibrium constitutional diagram of the aluminium-zinc system in Fig. 26. The temperatures at which loss of all strength occurred in each alloy are also included in this diagram. The temperatures of incipient fusion lie on a line some 20° C. below the solidus line of the equilibrium diagram. This effect is due to the severe coring which was present in the cast alloys.

The variation, with zinc content, of the brittle temperature range of the aluminium-zinc alloys is plotted in Fig. 27 and the results obtained by Pumphrey and Lyons on the cracking of aluminium-zinc alloys under conditions of casting and welding are plotted in Fig. 28. The amount of cracking encountered in restrained welds in the aluminium-zinc alloys is in close agreement with the extent of the brittle temperature range of the alloys as determined in the present work.

The tendency of aluminium-zinc alloys to crack in a casting test, however, has been found to decrease with increase in the zinc content above 6%. The decrease in the amount of cracking with increase in the zinc content above this figure was ascribed by Pumphrey and Lyons to the decrease in crystal size accompanying the increase in alloy content, and they postulated that, in the absence of eutectic in an alloy, the smaller the size of the crystals in the mass the more easily can they accommodate casting and solidification contraction by movement one upon another without the formation of cracks.

In order to examine the correctness of this suggestion a number of tensile tests were carried out during the course of the present investigation on two aluminium-zinc alloys, containing 16% and 20% of zinc, cast in such a way that they were of extremely fine crystal size. The temperatures at which incipient fusion and loss of all strength occurred in these alloys are plotted in Fig. 26. The extent of the brittle temperature ranges of the alloys is shown in Fig. 27 and it is apparent that the brittle temperature ranges of the alloys of fine crystal size are not appreciably less than those of alloys of the same zinc contents, but of larger crystal size. This result would seem to suggest that the reduction in the tendency of an alloy to crack with reduction in its crystal size is not due simply to a reduction in the brittle temperature range of the alloy.

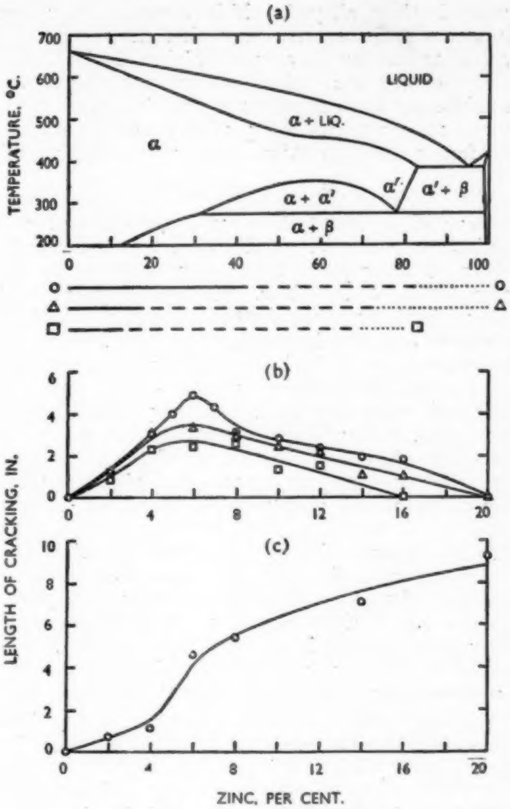

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Fig. 28.—Cracking of ring castings and restrained welds in the aluminium-zinc system (Pumphrey & Lyons). (a) Equilibrium diagram; (b) cracking of ring castings; (c) cracking of restrained welds.

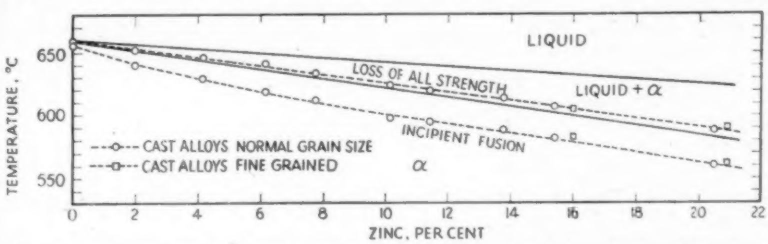

Fig. 26.—Temperature of incipient fusion and loss of all strength superimposed on a portion of the aluminium-zinc equilibrium diagram.

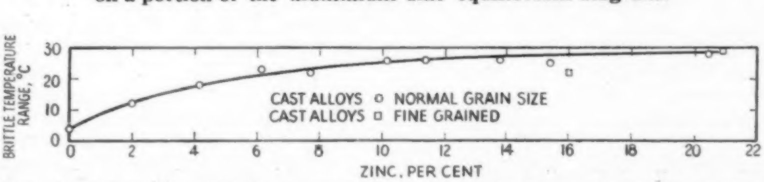

Fig. 27.—Variation of brittle temperature range with zinc content of aluminium-zinc alloys (derived from Fig. 26).

It is possible that the explanation of the discrepancy between the results of the ring casting and restrained weld tests, lies in a difference in the mode of solidification of the molten metal in the two tests. In the ring-casting test there is a greater mass of molten metal than in the restrained weld test, and the molten metal near the open upper surface of the casting is hotter than that near the mould wall so that crystals which form in the upper region of the casting, and which are not in an advanced stage of growth, can extend downwards into those regions in which "accommodation"[7] is occurring. This effect reduces the cracking which would normally occur at the surface of the ring casting but tends to leave intercrystalline

porosity in those alloys in which no eutectic is present.

It seems probable, therefore, that the results of the ring-casting test for the alloys of higher zinc content are to some extent misleading, because the alloys, whilst not exhibiting large amounts of cracking of an obvious nature, may yet not be perfectly sound and may suffer from a certain measure of intercrystalline porosity.

6. Conclusions

In a previous investigation by the present authors, an examination was made of the tendency of alloys in a number of binary aluminium-alloy systems to crack during casting and welding. The tendency to cracking of alloys in the systems aluminium-copper, aluminium-manganese and aluminium-zinc was found to be somewhat anomalous and in the present work an examination has been made of the tensile properties at temperatures above and below the solidus of alloys in these three systems and of alloys in the aluminium-iron system. An examination has also been made of the effect of the degree of approach to structural equilibrium on the high temperature tensile properties of certain of the alloys tested.

The tensile strength of all the alloys tested was found to decrease and the ductility, as measured by the percentage elongation and reduction of area, to increase with increase in the temperature of testing up to the temperature at which melting begins. With alloys containing little or no eutectic, the appearance of increasing quantities of liquid at temperatures of testing above that at which melting begins causes the strength and ductility to decrease, slowly at first and then more rapidly. While all ductility is lost at a temperature but little above that at which melting begins, a small strength is retained up to a temperature between solidus and liquidus at which so much liquid is present in the mass that all coherence and strength is lost.

With alloys containing a relatively high proportion of eutectic, a sudden fall in strength, accompanied by intercrystalline fracture, occurs at a temperature some 4° C. below the generally accepted temperature at which melting of the eutectic occurs in the system considered. This effect would seem to be due to melting occurring in the crystal boundaries at a temperature lower than that at which it occurs in the body of the crystals, an occurrence which may be ascribable to the physical nature of the boundaries, in particular to the high strain-energy in such regions, rather than to segregation of the alloying element or to the accumulation of impurities in the boundaries.

The effect of increasing degree of approach to structural equilibrium on the tensile properties at temperatures below the solidus of alloys in the systems aluminium-copper, aluminium-iron and aluminium-manganese has been examined, and found to be somewhat complex. The effect with the aluminium-copper alloys is to increase the strength, and with the aluminium-iron and aluminium-manganese alloys to decrease the strength, at temperatures below the solidus. The effect of increasing approach to structural equilibrium on the temperature at which melting first occurs in the alloys is to cause melting to begin at temperatures nearer the solidus temperature determined from the equilibrium constitutional diagram.

An attempt has been made to correlate the high temperature tensile properties of the alloys in the four systems with their tendency to crack during casting and welding, determined in a previous investigation. Good correlation has been obtained, in each of the four systems, between the curve relating the variation with alloy content of the range of temperature above the solidus in which a brittle fracture is obtained in the tensile tests and the curve relating alloy content with the tendency to crack during casting and welding. Further information has been obtained on the reasons for the anomalous behaviour of certain of the alloys in tests designed to determine their susceptibility to crack when cast or welded.

Acknowledgments

The work described in this paper was undertaken on behalf of the Aluminium Development Association and is published by permission of the Association.

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Automatic Welding of Structural Steelwork

FUSARC LTD. have designed and manufactured a large portal type automatic arc welding machine suitable for welding pre-fabricated H type and box section girders up to 100 ft. in length. The first machine, built specially to the order of Alexander Findlay and Co. Ltd. has been installed at their Motherwell Works.

Two Fusarc Automatic Welding Heads are arranged to run transversely on an adjustable runway; each head is provided with a separate control box and can be positioned and operated independently, or used simultaneously for longitudinal welding. Longitudinal traverse of the main structure is motorised for both high speed positioning at 16 ft. per minute and welding speeds infinitely variable between 7 in. and 28 in. per minute. An adjustable counterbalanced boom supports the cross traverse runway and, controlled from the operator's panel in conjunction with the head slides, allows a vertical operating height of between 1 ft. and 11 ft.

This installation is designed to tackle any straight line or rectangular work within the limits of the 100 ft. × 14 ft. floor space wherein it is at present operating, but the range of work could be increased by extending the supporting rails which are at floor level and by the addition of roller base, or tilting turntables.

Mullard Equipment Factory Expansion

To cater for the increased commercial activities of the Mullard Equipment Division, Mullard Equipment, Ltd., have recently acquired another factory at Wandsworth in Garratt Lane, London, S.W.18. All engineering development facilities, together with centralised workshop areas, are now being concentrated in the older factory at Brathway Road. At Garratt Lane are housed the main assembly and production lines for all classes of equipment, as well as factory administration and service departments. Research and advanced development will continue to be done at the Mullard Company's Research Laboratories at Salfords, Surrey.

The Corrosion of Steel under Phosphate Coatings and Protective Finishes

Report of Recent Conference in London

The use of phosphate coatings on steel as a basis for paint is one of the major developments in the struggle against corrosion. An account is presented here of the proceedings of a meeting held recently in London to discuss the efficiency of such coatings and the means of assessing this property.

THE efficiency of phosphate coatings on steel as protection against corrosion, and the means of testing this efficiency and corrosion resistance in general were discussed at a meeting in London on October 28th. The meeting was organised by the Iron and Steel Institute in conjunction with the British Iron and Steel Research Association, with Mr. H. T. Shirley, Chairman of the Corrosion Committee of B.I.S.R.A., in the Chair.

Papers for Discussion

Four papers were presented and discussed :

Phosphate Coatings as a Basis for Painting Steel. By the Phosphate Coatings (Drafting) Panel of the British Standards Institution.

Corrosion by Retained Treatment Chemicals on Phosphated Steel Surfaces. By S. G. Clarke and E. E. Longhurst.

Tentative Analytical Tests for Phosphate Coatings on Steel. By R. St. J. Preston, R. H. Settle and J. B. L. Worthington.

Second Report of the Methods of Testing (Corrosion) Sub-Committee.

The first three papers were discussed together. The first was presented by Dr. J. C. Hudson, the second by Dr. Longhurst and the third by Mr. Preston.

Phosphate Coatings as Paint Basis.

Dr. Hudson recalled that the initial purpose for which the Panel had been set up in 1944 was to consider tests of phosphate coatings and stoving paints for the Ministry of Works in connection with the large number of prefabricated houses then being built. B.S.I. Publication PD 539 of 1946 had resulted from the Panel's work ; this was really a code of practice, rather than a performance specification. It had proved useful, but the need remained for more specific tests, both analytical and performance.

The present paper described the results obtained when analytical and performance tests were applied to industrial phosphate coatings. Analytical tests included determination of :

(1) weight per unit area ;
(2) phosphate content ;
(3) moisture control ;
(4) hygroscopicity ; and
(5) absorption value.

The performance tests used were the A.R.E. seawater droplet test, the C.R.L. sulphur dioxide test and outdoor exposures.

The main difficulty met with in considering specifications was the wide variation between phosphating processes in current use, both in coating weight and PO_4 content. Table I shows some of these variations, as revealed by analytical tests on six types of industrial phosphate coatings. Each process, however, was satisfactory for the purpose for which it was intended, and field tests had, in fact, shown that wide variations in the weight of phosphate coatings applied by the same process had no effect on coating efficiency when overpainted.

TABLE I.
ANALYTICAL TESTS ON INDUSTRIAL PHOSPHATE COATINGS

Process	P	S	T	Q	V	R
Main cation in phosphate bath	Fe	Mn	Zn	Zn	Zn	Zn
Method of application	Immersion	Immersion	Spraying	Immersion	Immersion	Immersion
Duration of treatment, min.	15	30	1·5	4	5	12
Change in weight on phosphating, g./sq. ft	−2·40	−2·43	0·24	0·31	0·15	0·54
Coating weight, g./sq. ft.	1·31	1·95	0·41	0·50	0·32	1·13
PO_4, g./sq. ft.	0·64	0·82	0·18	0·19	0·11	0·41
Moisture, mg./sq. ft.	7·5	7	14	34	18	71
PO_4, content of coating, %	49	42	44	38	34	36
Moisture content of coating, %	0·6	0·4	3·4	6·9	5·0	6·4
Hygroscopicity of coating, %	0·3	0·2	1·2	1·3	1·0	1·5
Absorption value, g./sq. ft.	1·05	1·0	1·0	1·2	1·0	1·1

In spite of this, it might be possible to draw up specifications for individual processes, if each specification were strictly limited to the particular process concerned, and if the criteria of acceptance were suitably related to the purpose for which the phosphate coating was required. Such specifications might be based on a performance test for a specimen phosphated by the process under examination, and provided with a standard organic finish of oil, lacquer or paint. Experimental work to explore this possibility was in progress.

Dr. Hudson said that the B.S.I. Panel had studied the effect of chromic acid additions to rinsing water on two industrial phosphate coatings. Small concentrations of chromic acid had a beneficial effect on the protective properties of the coatings but a high concentration dissolved the coatings. The permissible limiting concentration of chromic acid probably varied for different phosphating processes, but even very dilute solutions with concentrations as low as 0·05% might have a beneficial effect. The optimum concentration was probably about 0·1 to 0·2%

Corrosion by Retained Chemicals.

Dr. E. E. Longhurst introduced the second paper on corrosion by retained treatment chemicals which, he pointed out, concerned hot bath phosphating only. The work was directed to answer the question : " How far is it necessary to wash the steel parts after phosphating ? " The answer varied, as might be expected, with the type of phosphating solution. In general, the risk of corrosion from solutions trapped in crevices or folds was less with the plain metal phosphate-phosphoric acid type bath than with accelerated phosphating solutions containing other constituents more active in stimulating corrosion.

Even with the plain type bath it was probably desirable to wash creviced parts after phosphating, though a relatively simple washing treatment to remove excess solution from recesses or from the mouths of crevices would be sufficient. With phosphating solutions containing other active constituents, washing was generally essential to remove residual corrosive solutions.

Dr. Longhurst supported these conclusions by a series of slides showing the condition of painted panels after test. He then suggested a simple performance test as a guide to the corrosive tendency of a solution if it was not known, or if the geometry of the parts treated made effective washing difficult. This test involved exposure of the parts to be examined to damp air for a period of one week in which period any dried out corrosive residues would tend to absorb moisture and become active.

Tentative Analytical Tests.

In introducing the third paper on analytical tests for phosphate coatings, Mr. Preston said that there were two possible lines of development. The first concerned the composition of the coating and the second concerned the coating's anti-corrosive qualities. Laboratory tests to determine the composition of the coating included the measurement of moisture content, phosphate content (for which two simple tests had been developed) and chromate content. Tests to indicate the anti-corrosive qualities of the coatings included absorption value, coating weight and hygroscopicity.

Mr. Preston said that the last-named was a most important factor. Accelerators used in industrial processes introduced salts which might attract water. For example, one firm of his acquaintance had encountered blistering due to salts contained in mains water ; the difficulty had been overcome by an alteration in the process.

Discussion

The favourable results obtained from plain types of phosphating bath by Dr. Longhurst were commented on by Mr. R. J. Brown (Morris Motors Ltd.). He said that these baths would be uneconomic in practice for many assemblies owing to the length of processing time. He suggested that the dip wash with very little agitation that Dr. Longhurst had employed might vitiate the comparison of his results with those from similar phosphate treatments which embodied a very efficient rinse with sprays.

Mr. Brown described how ring blistering of motor body finishes had been caused by water spots dropping on to clean body surfaces from overhead condensation points after phosphating and before painting. Even after continuous processing equipment had been introduced, trouble was encountered in the form of " snail trails " which were lines of fine blisters at the interface of the phosphate coating and the primer paint. An analysis of the rust associated with them showed the presence of calcium and chlorides. It appeared that incrustations of soluble salts in the overhead structure were being redissolved and were dripping on to the surfaces of the bodies. Further, one of the processing solutions used in the plant was acting as a source of chlorides. Correction of these two factors achieved a major reduction in " snail trails " but not their complete elimination from the plain body surfaces. The last source of contamination was found to be one which had been suspected quite early in the story ; the water supply used had a total hardness of 40 parts per 100,000. The volume of water consumed put out of the question the use of treated water such as boiler condensate, so that it was considered necessary to prevent localised evaporation of the water contained on the body surface. Sprinklers were used for this purpose.

The corrosion trails were still not completely eliminated but the final step was taken with the advice of the C.R.L. This consisted of the operation of the chromic rinse without heating and the prevention of rotation of the bodies (the cars are held on a spit during the process) between the chromic rinse and the forced drying. Uniform drying was then possible over the entire body surface without the formation of trails of soluble salts.

Mr. R. F. Drysdale (Walterisation Co. Ltd.) reported work broadly parallel in nature to that described by Dr. Longhurst but mainly concerned with lightweight coatings applied both by accelerated and non-accelerated processes. He had had the best results from specimens treated with non-accelerated processes which had been dried unwashed ; there was little to choose between those washed with water, chromic acid or soluble chromate solutions. Samples washed with a 0·03 molar phosphoric acid solution were comparable with those dried unwashed. In his view these differences in behaviour were associated with imperfect coating deposition within the seams owing to the poor circulation and, consequently, the exhaustion of the phosphating solution at such places.

Mr. A. Nicholson (Pyrene Company Ltd.) considered the question of rinsing generally. He thought that, even with unaccelerated processes, rinsing was advisable when the parts were to be subsequently painted. There would otherwise be risk of osmotic failure of the paint film. He suggested that a cause of failure at seams was incomplete coating formation. Cleaning inside the seam was the first and most difficult obstacle in this case. Accelerated processes, by virtue of their lower temperatures, strengths and treatment times, could be less active than unaccelerated processes in these particular conditions. Production considerations of time and space necessitated the use of accelerated processes and means were available to deal with seam problems if they arose. For example, he had known one case where seam trouble had been cured by *increasing* the accelerator concentration of the solution. He thought that a final rinse containing a small amount of phosphoric acid as well as chromic acid might be beneficial. The authors had mentioned a German patent concerning the use of a final rinse with zinc phosphate, which appeared to be comparable.

Mr. E. I. Brimelow (Building Research Station, D.S.I.R.) suggested there were dangers in assessing phosphate coatings by accelerated corrosion tests,

particularly to determine the most useful concentrations of chromic acid in the rinsing solutions.

Author's Reply

In reply to the discussion Dr. Hudson agreed with Mr. Brimelow that caution was necessary in such cases and re-emphasised that the optimum chromic acid concentration varied for different phosphating processes.

Dr. Longhurst replied commenting that Mr. Brown's interesting contribution related to a spray washing procedure, in which the car bodies being treated were never fully immersed and at times were fully exposed. Rapid evaporation had occurred from the hot exposed surfaces allowing concentration of soluble salts on the surfaces. These conditions varied considerably from those obtaining during treatment and washing by full immersion processes. He explained that in his own tests corrosion had occurred owing to the presence of certain constituents of residual phosphating solutions, and not by the action of added corrosive agents such as the salt spray in Mr. Drysdale's tests. He agreed that it appeared that small concentrations of phosphoric acid residues were not necessarily dangerous. In industrial practice, however, it was difficult to avoid major concentrations of phosphoric acid, owing to seepage from crevices or inefficient drainage on awkwardly shaped components. Minor defects would occur where such concentrations occurred, and generally he was in favour of a quick rinse in even the most favourable circumstances.

Replying to Mr. Nicholson, Dr. Longhurst said that in the work described by Dr. Clarke and himself, they had not studied the efficiency of phosphating within the crevices, but only the corrosivity of treatment solutions seeping on to the exposed surfaces outside the crevices. He confirmed that nitrate had been detected chemically near the crevice mouths on the specimens which had been phosphated by the accelerated process and subsequently washed.

Testing Methods Committee Report

Introducing the Second Report of the Methods of Testing (Corrosion) Sub-Committee, Dr. Hudson said that as a result of the Sub-Committee's work two laboratory tests for resistance to corrosion had been standardised and would shortly be published as B.S. 1391 : 1952. These were the A.R.E. salt droplet test and the C.R.L. test, which were based, respectively, on tests originally designed by the Armament Research Establishment and the Chemical Research Laboratory.

The development and standardisation of these tests had involved collaborative work in many different laboratories. In some respects the reproducibility of the tests was not all that might have been wished but it was generally as good as was to be expected in this type of experiment.

Dr. Hudson observed that reproducibility must be affected by the fact that any order of merit of a number of protective schemes could vary with the conditions of exposure, the type of service and the criterion on which failure was assessed. It was doubtful whether a laboratory test would ever give a hard and fast correlation with the results of service. Their best function was probably to ensure reproducibility of a given standard for a protective scheme of a given type. They were of great value in weeding out unsatisfactory protective schemes, but became fallible when it was a case of distinguishing between several good or excellent protective schemes.

In the Sub-Committee's attempts to adjust the criteria of failure so that a protective scheme would be equally likely to fail from general rusting or from the effects of damage, he thought that there might have been some tendency to exaggerate the effects of damage on performance. Often, although failure occurred at a damaged area, rusting remained localised and might even be stifled. In the new British Standard B.S. 1391 : 1952 therefore, the infliction of damage before testing had been made optional, so that a user expecting damage to be the determining factor under his particular service conditions, could use the standard methods of damage before the tests. Otherwise, the tests could be carried out on undamaged specimens.

The Queen Accepts Lead Medal

This year marks the centenary of the Derbyshire Mining Customs and Mineral Courts Act. This Act legalised in 1852 the traditional rules and customs of the medieval Great Barmote Court for the Soke and Wapentake of Wirksworth. The known records of the Court date back to the 13th century, but there is very little doubt that it was in existence in some form during the Roman Conquest and Saxon Settlement. It has acted on behalf of the Duchy of Lancaster in the settlement of claims and disputes between miners within the King's Field in Derbyshire for at least 700 years. Nowadays its Officers and Jurymen still sit twice a year to fulfil the Court's time-honoured duties.

This year's session is rendered particularly noteworthy by the fact that Her Majesty the Queen has graciously consented to accept a medal specially struck to commemorate these ancient rights of the lead mining community in Derbyshire. It was cast, appropriately enough, in lead mined in Derbyshire by the Derbyshire Stone Company. The refining of the lead was undertaken by Messrs. H. J. Enthoven & Sons, Ltd., one of the most prominent lead refiners in the country, at their Darley Dale Smelter.

This is the first time since the accession of Maria Theresa in Austria in 1743 that a lead medal has been struck in this country.

NEWS AND ANNOUNCEMENTS

The Institution of Mining and Metallurgy

THE Council of the Institution of Mining and Metallurgy have great pleasure in announcing that His Royal Highness The Duke of Edinburgh has graciously accepted election to Honorary Membership of the Institution.

Forty Foot Aluminium Cutter

By its interest in the use of light alloys, the yacht building world has provided a nursery for what is likely to be a large scale development in commercial shipbuilding. It is a small step from the larger yachts to the smaller trawlers and coasters amongst which the next stages of this evolution may be expected.

The *Coimbra*, designed by Laurent, Giles & Partners, Ltd., and built by Hugh McLean & Sons, Ltd., for Mr. N. B. Redfearn, a South African, is a 40-ft. ocean going yacht fitted with a 10-24 h.p. Parsons Ford petrol engine as auxiliary power. In her case, a mean has been struck between the reduced displacement and increased carrying capacity made possible by the use of light alloys, and, with an economical speed under power, either alone or sail-assisted, of between 5 and 6 knots, her range is virtually unlimited

G.W.B. Induction Melting Furnaces

G.W.B. ELECTRIC FURNACES, LTD., of Dudley, Worcs., have entered into an agreement with S.P.A. Forni Elettrici A. Tagliaferri, of Milan, to market normal frequency core type induction melting furnaces. The range includes standard furnaces for melting aluminium and other light alloys, copper alloys and cast iron. In addition, furnaces are available for zinc distillation in a closed furnace chamber. The melting is carried out in a channel surrounding a primary winding, a ring of molten metal forming the secondary winding of a 50 cycle transformer.

The melting furnaces for cast iron have been specially developed to work in conjunction with normal cupolas in the duplex process for producing high grade refined irons. Such furnaces can also be used for producing nickel-manganese and spheroidal graphite cast irons, whilst cast iron chips and scraps can be recovered in these induction furnaces with a far smaller metal loss.

All furnaces are arranged for either hand, mechanical or hydraulic tilt and, in the case of the larger types, a special feature lies in the ability to remove the bath and inductor slots for refining without disturbing the transformer itself or the electrical connections. This means that a spare bath can be held in readiness, relined for immediate use.

West African Aluminium

A £144 million project for developing production of aluminium in the Gold Coast to an ultimate rate of some 210,000 tons a year was outlined in a Government White Paper published recently. This scheme, to be known as the Volta River Aluminium Project, will result in a partnership between private enterprise and the U.K. and Gold Coast Governments, the former being primarily responsible for aluminium output and the latter for a big hydro-electric scheme and public works and services.

Driver-Harris Conference

AT the Fourth International Gathering of Associates and Distributors of the Driver-Harris Companies, the improved Driver-Harris alloys and their greatly expanded fields of application in many countries of the world were surveyed. A major part of the conference was devoted to a discussion of seven papers: (*a*) the new nickel-chrome alloy "Karma"; (*b*) "Nichrome" and "Nichrome" V developments; (*c*) nickel-iron alloys; (*d*) sparking plug alloys; (*e*) nickel alloys for electronic and electrical uses; (*f*) material for water meters; (*g*) thermocouple materials.

The conference, attended by 50 delegates from several European countries and from the U.S.A., South Africa and the Argentine, was welcomed by Mr. R. M. Parry, Managing Director of British Driver-Harris Co., Ltd.,

A group of delegates to the Technical Development Conference at the 4th International Gathering of Associates and Distributors of the Driver-Harris Companies.

Manchester. The programme included a half-day tour of the greatly extended factory at Cheadle, Cheshire.

During a discussion on patents and registered names, it was stated that there was a danger of the most important of the latter—"Nichrome"—becoming a generic term, and that every available step had been taken, throughout the world, to establish on the firmest ground the fact that "Nichrome" is the registered trade-mark of the Driver Harris Companies.

Aluminium Production Film

REFERENCE was made in our columns earlier this year to a film on aluminium production which won the award for the best industrially sponsored film made in Canada in 1951. This 32-minute feature in colour—"Packaged Power"—is now available, through Northern Aluminium Co., Ltd., for showing to colleges, schools and general audiencies in this country. The film tells the story of aluminium seen through the eyes of the Aluminium, Ltd., Group of Companies, and in so doing gives an account of an important industrial enterprise within the Empire. Explaining by means of photography and animated drawings the process by which aluminium is extracted and fabricated, the film moves between British Guiana, Canada and Great Britain, concluding with a review of vast new projects for the mining and treatment of the ore in Jamaica and its reduction in British Columbia.

The Fight Against Rust

THE work of the Corrosion Committee of the British Iron and Steel Research Association, which was formed in 1928 by the Iron and Steel Institute and taken over by B.I.S.R.A. in 1945, is described in a 25-page booklet published by the Association at 2s. 6d. post free.

The account is presented under the following headings: case histories; applied research; fundamental research; summary and practical conclusions. The first two subjects deal with corrosion by air, water and soil, seven case histories being discussed. Systematic exposure tests and conclusions from them are described, together with work on protective coatings and methods of testing. Summaries of work at Cambridge University and the Chemical Research Laboratory are also given.

Anodic Protection of Ships

THE use of magnesium in the anodic protection of buried water pipes is well known to those interested in the subject of corrosion. The method has now been applied to the protection of ship's hulls and successful results are claimed for it after a years experience on the tanker *Marine Chemist* which was drydocked recently at the Bethlehem Steel Company's yard at Brooklyn. The corrosion control system consisted simply of magnesium rails attached by steel studs to the sides of the hull.

Price Pegging

KEITH BLACKMAN, LTD., announce that, in spite of the recent engineering wage increase award, and of subsequent rises of raw material and component prices, they have decided to attempt to absorb, at least for the present, the whole of the increased costs. It is pointed out, however, that it will be necessary to maintain production at the highest level.

Personal News

DR. F. A. FOX has joined the Australian Government Department of Supply as Superintending Scientist in charge of the Department's Chemical and Physical Research Laboratories, Maribyrong, Melbourne. His address, after January 4th, 1953, will be:—c/o Department of Supply, 339, Swanston Street, Melbourne, Australia.

MR. J. E. C. BAILEY, C.B.E., Chairman and Managing Director of Baird and Tatlock (London), Ltd., and of Hopkins and Williams, Ltd., is planning to visit Africa in January and intends, during his two months' stay, to contact scientific establishments in Khartoum, Kampala, Entebbe, Nairobi, Mombasa, Dar-es-Salaam, Zanzibar, Lusaka, Salisbury, Johannesburg and Durban.

MR. L. M. C. ROBINSON has been appointed Naval Architect to the Aluminium Development Association in place of MR. J. VENUS, who has resigned to take up an appointment with a firm of consultants. Mr. Robinson was until lately Assistant Shipyard Manager (Neptune Yard), Swan, Hunter & Wigham Richardson, Ltd. Previously he had been for a number of years with the Royal Corps of Naval Constructors, Admiralty, at Bath.

GARRINGTONS, LTD., Bromsgrove, Worcs., announce the appointment to the Board of MR. G. F. BROWN (Secretary), as Director and Secretary, and MR. B. E. J. HODSON (General Works Manager), as Works Director.

MR. MAX DAVIS, Public Relations Officer of the British Iron and Steel Federation has been elected to the Council of the Newcomen Society.

MR. H. SILMAN is leaving Joseph Lucas (Industries) Ltd., to take up an appointment as Research Manager to the Ford Motor Co., Ltd., who are setting up a research organisation in Birmingham.

MR. W. C. FAHIE announces that he is no longer acting for Tinsley Industrial Instruments, Ltd., North Circular Road, London, N.W.10.

Obituary

WE regret to record the deaths of the following:—

MR. ROLAND FINCH, a personality in the metal trade for many years, who died in Birmingham on November 24th. When he retired in 1946, Mr. Finch was a joint Managing Director of I.C.I. Metals Division. After graduating in mechanical engineering at the City and Guilds of London College, he joined Kynoch, Ltd., in August, 1902, as an assistant to the general manager. Thus began an association with Kynoch's and, later, associated firms in I.C.I. which was to last for more than 40 years.

MR. FRANK GURNEY, who died in a London hospital on November 27th after a brief illness. Mr. Gurney, whose appointment as Home Sales Manager of Metropolitan-Vickers Electrical Co., Ltd., was announced recently, had been with the Company for over 30 years. After a brief period in Motor Sales Department he spent a number of years in the Company's district offices, leaving the managership of the Leeds office in 1941 to return to Trafford Park to become Sales Manager of the Transformer Department, and, four years later, Sales Manager of the Plant Department. He was appointed Assistant Manager Home Sales in 1949.

RECENT DEVELOPMENTS
MATERIALS : PROCESSES : EQUIPMENT

Automatic Sliding Doors for Factories and Warehouses

Doors developed primarily for lift entrances by The Express Lift Co. Ltd. have been adapted to provide automatic sliding doors suitable for factories and warehouses. Because the doors close automatically they prevent draughts and facilitate the maintenance of a constant temperature within the shop, thus ensuring maximum benefit from the consumption of fuel for space heating.

The doors consist of two sliding panels opening from the centre and run on ball bearing rollers on bright steel tracks. They are opened and closed by an electric operator which, to prevent excess torque being transmitted to them, incorporates a clutch. Fitted to the doors is a sensitive edge that ensures their instant reversal should an obstruction be encountered. An adjustable time delay is embodied in the controller so that the doors are held open for any pre-determined period within the limits allowed. The doors are opened by means of press buttons which can be fitted at any convenient point.

The General Electric Co. Ltd., Magnet House, Kingsway, London, W.C.2.

Specific Gravity Balance

Stanton Instruments, Ltd., announce the availability of a new specific gravity balance of the Westphal type, which was first shown at the recent International Congress on Analytical Chemistry at Oxford.

The balance has a 10-in. lacquered beam fitted with a separate rider bar with 100 accurate notches, numbered divisions being from zero on the left to 10 on the right. Specific gravities of liquids ranging from 0 to 2·0000 can be determined to an accuracy of 0·0001, with the use of only two riders and one range weight, all of which are made of stainless steel. One of the principal features is that the balance incorporates an arrestment mechanism with front release, as used in conventional analytical balances. Knife edges and planes are of selected agate. The instrument has an accurately adjusted plummet and an adjustable platform so that vessels of different shapes and sizes can be used. It is fitted on a board with levelling screws and spirit level and an outer case can be supplied if required. Accessories for the testing of solids and for the determination of specific gravity of liquids beyond 2·0 are also available.

Stanton Instruments, Ltd., 119, Oxford Street, London, W.1.

Die Polishing Machine

An addition to the range of equipment manufactured by Rudkin and Riley Ltd., for the treatment of wire, rod and tube drawing dies, is their type 2006/3 Hand Polishing Machine. The machine is fitted with $\frac{1}{2}$ h.p. motor, starter and isolator mounted on a common base, to form a complete unit ready for wiring to the electric supply.

The quick-release hand rest, which is shown in the illustration, is normally fitted for die-work and is designed for instantaneous positioning by the operator. Its use acts as a safeguard and as a steady to the operator when pressure is applied to the workpiece, particularly when blending the radii. A 4 in. self-centring chuck is fitted as standard but the robust design of the headstock, incorporating liberally-rated bearings, enables larger chucks (maximum 8 in.) to be fitted where required. Grease nipples in the headstock are conveniently located for lubrication, and a double-grooved pulley provides alternative polishing speeds of 2,400 and 3,150 r.p.m.

A reciprocating hand-tool which is also manufactured by the Company, and assimilates the hand polishing operation but at greatly increased speed, is most suitable for use in conjunction with this machine. In addition to drawing dies, polishing operations on spindles, guides, bushes and other circular workpieces may be carried out on this machine.

Rudkin and Riley, Ltd., Cyprus Road, Aylestone, Leicester.

CURRENT LITERATURE

Book Reviews

THE STORY OF MOND NICKEL

THIS excellent book, written to mark the Jubilee of the Mond Nickel Company, is both interesting and informative. It presents a brief record leading to, and from, the formation of the company and includes sketch-portraits of some of its past leaders. The discovery that nickel and carbon monoxide could be made to combine was announced in a paper, presented to the British Chemical Society in 1890 by Ludwig Mond, Carl Langer, and Friedrich Quincke. Although the existence of nickel in almost every explored part of the world had become known, it was regarded as a comparatively rare metal, and the difficulties associated with its extraction were such that it is doubtful whether a large market could have been supplied, even had it existed. Although the paper referred to described the use of nickel as a means of removing carbon monoxide from a mixture of hydrogenous gases, Mond quickly recognised the possibilities of the discovery when he experienced difficulty in producing a similar reaction with other metals, and he applied for a patent covering the manufacture of nickel.

Just previously, Riley had presented his famous study of the properties of nickel steel. He demonstrated that nickel conferred on steel qualities of strength and toughness hitherto unknown, the significance of which was not lost on Mond and his collaborators who, despite numerous difficulties, took immediate steps for the practical exploitation of the discovery. In 1892, by arrangement with Henry Wiggin and Company a complete experimental plant was built at Mond's expense on part of their land at Smethwick. Doubtless the fact that Henry Wiggin and Company were refiners of nickel influenced the choice of site.

While the technical processes were mastered, other problems remained formidable. Mond saw that it was not enough to evolve a new and unique process ; he had to extract and market a substantial quantity of nickel if the process was to become an economic success. The possibility of disposing of his patent rights was explored, but by 1898 negotiations were broken off and efforts made to acquire ore properties in the Sudbury region. Property was eventually taken over and a mass of detail relating to the building of railways and sidings, purchase of rolling stock, timber, mining machinery, crushers, boilers and so on, were dealt with and it was decided to obtain in Canada plans for a smelter. In 1900 was started the erection of a refinery at Clydach, near Swansea, and subsequently a public company was formed to take over Mond's processes and properties. A subscription list was opened about the middle of 1901, for a capital of £600,000. For the business, patents—there was seventy of them—and properties, Mond received £325,000, having from first to last expended more than four-fifths of this sum from his own pocket.

Thus in about ten years of research and experimentation, of planning and bargaining, of buying and building, an idea had become a material fact. Mines, smelter and refinery were linked in an organisation which gave them life and caused them to operate and produce—nickel. The treatment of matte began early in 1902 and the first deliveries of metallic nickel and copper sulphate in the summer of that year. It is a tribute to the skill and foresight then displayed that for fifty years the essential features of the main equipment and process have remained unaltered.

The Company, which was founded on research and experimentation, has continued to apply further scientific knowledge by similar though intensified means, and has reached even higher levels of achievement. Its growth and progress, however, have been due, in a large measure, not only to an untiring quest for further scientific knowledge, but also to an unfailing readiness to see, and to take advantage of, the wider industrial opportunities which that increasing knowledge gave.

Although this book contains only 63 pages, it is admirably written by Mr. A. C. Sturney and illustrated by Mr. Eric Fraser and, in addition to the early struggles of the Company, it deals with developments from 1914 to 1928, with the agreement negotiated in 1929 whereby the interests of The Mond Nickel Company were merged into The International Company of Canada, and with the more recent war achievements. The book has been printed privately for The Mond Nickel Company.

STRENGTH OF MATERIALS

By Arthur Morley. 10th Edition. 583 pp., index and appendices. Longmans Green and Co. Ltd., London, 1952. 25s.

Although there are many good text books on Strength of Materials, Dr. Morley's book is probably the most widely known. It was originally intended for students in Universities and Technical Colleges taking examinations of degree standard, but has since become a very valuable addition to the libraries of industrial firms, and few stress and design offices fail to have a copy on their shelves.

Chapters I to XIV, inclusive, remain unchanged in the tenth edition, apart from minor corrections, and whilst the method of presenting the subject matter and the derivation of the equations has always been extremely clear and needs no revision ; it is, perhaps, a little disappointing that an opportunity has not been taken of bringing the various references to research and materials up to date. For instance, in dealing with the present knowledge of fatigue phenomena in Chapter III, no reference is made to the large amount of work which has recently been carried out in this field, particularly with light alloy components. The book adequately covers the syllabus for engineering degree courses, and there is much additional information which is as useful to people engaged in industry, but its value to the aircraft engineer would have been enhanced had it included an introduction to the theory of the ultimate strength of members in bending, with a definition of the form factor and a description of the deformation of the member in the plastic state.

A useful introduction to the highly specialised subject of determining stresses in components by means of

strain gauges is now given in Chapter XV. An analysis of the results obtained from strain gauges is adequately covered in a new appendix and is important as a thorough grasp of the principles mentioned is essential in view of the wide use now made of this method of stress analysis. The inclusion of a further appendix dealing with close-coiled helical springs when the ratio of coil diameter to wire diameter is small fills a gap left in previous editions and will be profitable to students and engineers.

The tenth edition remains what the book has always been, an admirable text book on strength of materials and one which is invaluable to all who deal with engineering materials.

WÖRTERBUCH DER SCHLEIF- UND POLIERTECHNIK

By Senator N. C. B. Kleinschmidt. 96 pp. 25 illustrations, 1952, Berlin W35. Technischer Verlag Herbert Cram. DM 9.80

In every industry, many idioms or colloquial terms are used which are not to be found in an ordinary dictionary. The work of the translator is further complicated by the fact that there is considerable variation in many of these special terms as applied to a given process, material, tool, etc. Consequently, the practical value of a technical dictionary depends largely, apart from its general scope, on the number of important colloquial terms it contains, and upon the accuracy of their definitions.

It is claimed for this German–English, English–German dictionary, which has been produced to serve the abrasive engineer and the user of grinding and polishing materials, that it contains all the grinding and polishing terms which, on account of their novelty, have not yet been taken up by other technical dictionaries.

The book is printed on good quality paper but its appearance is marred in places by imperfect alignment of the type. There are instances, too, of mis-spelt English words, such as " ferreous " for " ferrous " and " general shope use " for " general shop use," whilst the American influence is to be seen in the spelling of words ending in -tre : " centre " becomes " center," " lustre " " luster " and so on. These are minor criticisms, however ; no doubt the book will be of considerable service to those engaged in grinding and polishing, whether German or English, who have cause to read foreign technical publications on the subject.

Trade Publications

" Conductor Accessories," Publication No. OT.12 of the Aluminium Wire and Cable Co. Ltd., has been issued with the object of listing and describing accessories supplied by the Company for use with steel-cored aluminium, all-aluminium and Silmalec overhead conductors. Wherever necessary, the method of application of each accessory is described, and attention is directed to points of special importance. The correct size of accessory to be used with any particular size of conductor is stated in the appropriate table. Copies may be obtained from 37, Thurloe Street, London, S.W 7.

We have received from Refractory Mouldings and Castings, Ltd., a copy of their new catalogue " Refractory Shapes" which deals with the complete range of the Company's products. These include tailor-made shapes in a range of materials based on fireclay, molochite, sillimanite, silicon carbide, alumina and zircon-zirconia. The method of manufacture, without the addition of clay, gives the advantage of higher temperature ranges, the maximum working temperatures varying from 1,200° C., for the fireclay-base type, to 2,000° C. for the zircon-zirconia base type. R.M.C. products are not intended to compete in price with the general run of refractory squares and firebricks—their fields of application are where intricate shapes or other factors make accuracy of dimension, sound structure and freedom from impurities desirable.

The latest ALAR data sheet describes the properties and characteristics of the low expansion alloy to specification B.S. 1490-LM-13. Related American specifications include A.S.T.M. B 179–49 TSN 122A, SAE 321 and Alcoa A 132. Copies may be obtained from Alar, Ltd., 3, Albemarle Street, London, W.1.

The Industrial Group of Philips Electrical, Ltd., has recently issued its first combined Industrial Catalogue (PD 3444). This consists of 32 pages concisely setting out the full range of Philips' industrial products under section headings, such as arc-welding, resistance welding, electronic instruments (no fewer than 78 are listed), high-frequency heating, etc. The leaves are loosely bound in a cover which permits prospective additions and alterations to be made at a later date. Copies may be obtained from Century House, Shaftesbury Avenue, London, W.C.2, or from Branches and Depots.

The latest General Refractories leaflet concerns the G. R. Sillmax Bricks introduced to meet a need for a higher grade brick of the aluminous type to withstand severe operating conditions in modern oil-firing and other installations. Details are given of the properties of the four grades available together with suitable applications for each grade. Copies may be obtained from Genefax House, Sheffield, 10.

We have received from Keith Blackman, Ltd., Mill Mead Road, Tottenham, London, N.17, a copy of the recently published catalogue, Publication No. 23, giving details of Tornado paddle wheel type fans, which command a wide field of application in connection with dust and fume removal, pneumatic conveying and other work. The unrestricted air passage renders the paddle wheel type fan unequalled for dealing with materials of a clogging nature.

In Automatic Controller Catalogue R/30, Negretti and Zambra, Ltd., list the range of instruments and accessories available for controlling industrial processes This hundred-page publication gives details of automatic controls for temperature, pressure, humidity, liquid level and specific gravity, together with all the valves, motors, relays, alarms and switches necessary to work in conjunction with them.

" Taking Care of Bearings " is the title of a workshop manual issued by Fischer Bearings Co Ltd., Upper Villier's Street, Wolverhampton. The Company's view is that, as great care is exercised in the production of its bearings, and every effort is made to ensure that material, workmanship and accuracy maintain a high standard, it is also necessary that users of bearings should exercise the same degree of care in fitting and maintenance, as careless treatment at this stage may result in a bearing failure.

LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC

INSTRUMENTS AND MATERIALS

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Recent Developments in Metallurgical Analysis
I.—Electrochemical Analysis

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Many advances have been made in analytical methods during the past few years. The present series of articles reviews some of these developments under the general headings: I—Electrochemical Analysis; II—Titrimetric Analysis; and III—Gravimetric Analysis. Such is the volume of papers forthcoming in those three main divisions, however, that it is only possible to touch on the more interesting developments which have appeared within the past few years, although references have been made to work previous to this period where it has been felt necessary to trace the logical development of some of the methods.

THE possibility of applying an electric current in metallurgical analysis was realised and used in a qualitative manner in the opening years of the nineteenth century, but the first quantitative work was not carried out until mid-century, when Wolcott and Gibbs initiated the technique of electro-analysis. Since then there has been considerable activity in the field, and the method has achieved widespread popularity.

Salient points in the development of this analytical tool are the evolution of the mercury cathode by Smith in 1903 and the introduction of rotating platinum electrodes by Sand in 1907. The possibilities of controlled electrode potential electrolysis were demonstrated at an early date, but it was not until the introduction of automatic control devices that this important development received recognition.

The simple technique of internal electrolysis, which dispenses with the use of applied voltages and currents, is likewise receiving more attention, particularly in methods which necessitate the determination of trace metals in many modern alloys. The first work on internal electrolysis was carried out by Spencer, Jacobi and Jordan in 1839. Sand[1] introduced an important modification by speeding up the analysis through vigorous stirring, and the use of an elevated temperature. In this way determinations have been made of bismuth and copper in bullion, and of copper, bismuth, cadmium and silver in ores[2].

Diehl[3] has published a monograph on the technique of controlled cathode potential electro-analysis, and has given a detailed discussion of the determination of silver, bismuth, copper, lead, antimony, tin, cadmium and nickel. The analysis of bearing alloys, brass, and bronze is fully dealt with.

Another means of using the electro-analytical method in metallurgical analysis comes in the technique of coulometry. In this case the deposited metal is not weighed, but the current passed is related to the amount of material that must have deposited on the electrode during analysis. The pioneers of this work in metallurgical analysis were Szebelledy and Somogyi[4] who electrolysed at constant current with platinum electrodes till a suitable indicator in the electrolysis medium showed the end of the operation. Other workers prominent in this field are Zakharevskii, and Hickling and Lingane.

Electro-analytical methods are particularly useful for effecting the separation of one metal from another. In many cases the electrolysis method is more efficient and much less involved and tedious than the classical chemical methods, and requires relatively simple apparatus. The deposition of metals is generally rapid and quantitative, and the method can be applied over a wide range of concentrations with the minimum of modification. Electro-deposition is readily placed on a fully automatic basis, and is therefore easily adapted to routine test analysis.

The Determination of Copper in Brass, Bronze and Tin Base Alloys, etc.

The separation of copper and tin may be effected by electrolysis from media containing phosphoric and nitric acids[5], or nitric and hydrofluoric acids[6,7], but the subsequent determination of tin in the electrolysis solution requires the removal of the nitric acid by fuming with sulphuric acid. A recent development in the analysis of brass and bronze by Norwitz[8] has shown that this difficulty may be overcome by carrying out the electrolysis from strong sulphuric acid. The only drawback recorded by the author is the fact that small amounts of antimony are quantitatively deposited with the copper.

A 1 g. sample of the brass or bronze was dissolved in 20 ml. of 70% nitric acid by boiling on a hot plate: 25 ml. of 97% sulphuric acid were added and the solution was evaporated and fumed for a few minutes. After cooling, 150–175 ml. of water were added and the lead sulphate allowed to settle out for a few hours. The copper was electrolysed at 2 amps./sq. dm. for 1 hour, using a platinum gauze cathode and a platinum spiral anode, stirring well during electrolysis. The tin was determined titrimetrically in the usual manner by addition of hydrochloric acid and the use of aluminium as reductant.

The antimony in bronzes ($< 1\%$) was corrected for, after weighing, by redissolving the deposited metals in 50% nitric acid with addition of phosphoric acid. The oxides of nitrogen were boiled out of the solution, one drop of 0·1 N hydrochloric acid added and the copper electrolysed, the weight of antimony being found from the difference in weights. The method was not suitable where it was necessary to separate the copper from amounts of antimony in excess of 0·25–0·3 g. Above this level, contamination of the copper resulted in high analysis figures.

The electro-analysis of copper in tin-base alloys was effected by solution of the alloy in a mixture of hydrochloric, nitric and phosphoric acids[5]. The excess nitric and hydrochloric acid was removed by digestion for half an hour on the hotplate. Pyrophosphoric acid was converted to phosphoric acid by boiling with water for 10 minutes, nitric acid added, and the copper deposited. Norwitz[9] later improved this method by using hydrogen peroxide and eliminating the necessity of heating and digesting the solution. In the modified procedure a 1 g. sample of tin-base alloy was dissolved in 10 ml. of 48% hydrofluoric acid, 10 ml. of water and 10 ml. of nitric acid, without heating: 5 ml. of 85% phosphoric acid were then added and 25 ml. of 3% hydrogen peroxide (10 vol. H_2O_2). The solution was diluted to 200 ml. and electrolysed at 2 amps./sq. dm. for 15 minutes using platinum electrodes. When electrolysis proceeded beyond 20 minutes contamination of the deposit occurred. The method is not suited to the analysis of tin-base alloys containing silver or bismuth in excess of 0·5%, but an arsenic content less than 5% can be tolerated. Altogether the method is an elegant one, and since sample preparation requires only 2 minutes, and the electrolysis 15 minutes, it has much to recommend it.

Kovalenko[10] reported on the use of an oxidised aluminium cathode for the determination of copper in the presence of small quantities of cadmium, and Goldberg[11] has developed a rapid method for the electro-analysis of copper and lead in a silicon aluminium bronze alloy. Norwitz[12] has effected the electro-analysis of copper in aluminium alloys containing tin and antimony by using a phosphoric acid medium, and in the presence of bismuth and arsenic using hydrogen peroxide in the electrolyte[13,14]. He has also reported a method in which arsenic and copper are deposited simultaneously from a solution containing hydroxylamine hydrochloride, the arsenic being present as copper arsenide[15]. Veitsblit[16] has recently studied the determination of copper in the presence of tin, using a hydrofluoric acid medium.

Determination of Silver and Separation from Copper

The same author[17] has also published a new method for the determination of silver and its separation from copper. Methods for the electro-analysis of silver are generally unsatisfactory unless the metal is being deposited from pure solution after a preliminary separation from other metals. Thus, for example, the cyanide method is excellent for pure solutions of silver, but cannot be used satisfactorily in the presence of copper unless a rather low current density is used[18]. Lead and bismuth are co-deposited with the silver. The use of a nitric acid medium is unsatisfactory because of the non-adherent nature of the deposit on the cathode[19]. Copper and bismuth interfere unless the cathode potential is carefully controlled. Better results may be obtained in nitric acid medium by use of a mercury-coated platinum cathode, but the drying of the silver amalgam without loss of mercury is a difficult operation. A nitric acid medium containing ammonium acetate gives good results with pure silver solutions, but again copper and bismuth interfere unless there is careful control of the potential at the cathode; the same remarks apply to deposition from ammoniacal solution. Addition of peroxide to the ammoniacal solution yields a satisfactory separation from copper, but under these conditions lead, nickel and bismuth interfere[20].

Norwitz's method involves deposition from a nitric acid medium containing sodium nitrite; the deposit is bright and adheres to the electrode. Neither the amperage nor the voltage is critical, and there is no tendency for silver oxide to deposit on the anode. Bismuth, copper, lead, nickel, cobalt, cadmium, zinc, iron, chromium, manganese and aluminium do not interfere. Subsequently the nitrogen oxides may be boiled out of solution and the copper may be electrolysed in the usual manner.

The sample was dissolved in 20 ml. of 50% nitric acid by warming on a hotplate and the solution diluted to 190 ml. with addition of 6 g. of sodium nitrite. Electrolysis was carried out for 1 hour at 2 amps./sq. dm. Excellent results were obtained for amounts of silver varying from 0·5–0·1 g. in the presence of amounts of copper varying from 0·5–0·1 g.

Determination of Cadmium

Osborn[21] has recently evolved a method for the determination of cadmium in the presence of nickel and zinc, and published details in this journal in 1949. By electrolysing from a dilute solution of sulphuric acid or perchloric acid in the presence of gelatin, a hard, clean deposit of cadmium was obtained. Simultaneously a method was described for the determination of both metals when the solution contained only cadmium and zinc. The use of gelatin was also recommended by Zivanovic[22] in his method for the analysis of cadmium in zinc dust and zinc concentrates. In this case the sample was dissolved in a mixture of hydrochloric and nitric acids and brought down to fumes with sulphuric acid. Gelatin and boric acid were added after removal of other interfering ions, and the cadmium was deposited on a platinum cathode, using a zinc anode at a temperature of 80° C.

The Determination of Selenium and Tellurium and their Separation from Copper

Most of the methods published for the electrolytic determination of selenium and tellurium, and for their separation from copper, are time consuming and require very close control of amperage or voltage. When the critical voltage or amperage is exceeded the selenium and tellurium precipitate from solution and unsatisfactory separations from copper may result.

No method for the electro-analysis of selenium involving the deposition of metallic selenium is known. Müller[23] described an electrolytic method dependent on the deposition of copper selenide on a cathode from 2 N sulphuric acid, while Jilek and Lukas[24] determined selenium by deposition of cupric selenide and bismuth selenide from a nitric acid medium containing sodium tartrate. In both cases it was necessary for the selenium to be in the quadrivalent state before the start of the

electrolysis. Tellurium on the other hand can be determined by deposition in the metallic state as, for example, in the methods of Pellini[25], and Gallo[26] where deposition was effected respectively from a medium containing ammonium tartrate and from sulphuric acid containing sodium pyrophosphate. Müller[23] deposited elemental tellurium from 2 N sulphuric acid at a cathode potential in the region 0·04–0·10 V. Again it was necessary that the tellurium should be present in the quadrivalent state in all these methods.

To separate the two metals from copper, however, it is necessary in all cases that they be in the hexavalent state (i.e., they must be present as selenates or tellurates). Smith[27] separated copper from selenium by using a cyanide medium. He also recommended a nitric acid medium with a current density of 0·05–0·08 amp./sq. dm. and 2·0–2·5 V., and a sulphuric acid medium at 0·05–0·10 V.

Smith [27] separated copper from tellurium by use of a sulphuric acid medium at 0·05–0·10 amp./sq. dm. and 2–2·25 V. Newton and Furman[28] separated copper from selenium and tellurium in a nitric acid–sulphuric acid medium and also in a mixture of nitric, sulphuric and hydrofluoric acids. The selenium and tellurium were oxidised to the hexavalent state by means of persulphate, and the voltage was carefully controlled at 2·1 V. for the separation of copper from selenium, and at 2·0 V. for the separation of copper and tellurium. The time necessary for separation varied from 5 to 16 hours. The amount of selenium that could be separated from copper was 0·05 g. from sulphuric acid and nitric acid, while the amount of selenium and tellurium that could be separated in the mixture of nitric, sulphuric and hydrofluoric acids was approximately 0·15 g.

Norwitz[29] has developed a much more rapid method in which selenium and tellurium are deposited and weighed as copper selenide and copper telluride from fairly strong nitric acid, containing the selenium and tellurium in the quadrivalent state. A method was also described for the separation of selenium and tellurium from copper in the same medium after oxidation of the selenium and tellurium to the hexavalent state with permanganate. The maximum amounts of selenium and tellurium determined by Norwitz were 0·25 g. and 0·017 g. respectively. Copper was separated from 0·045 g. of selenium and 0·15 g. of tellurium.

The Determination of Thallium and Lead

The electro-analytical determination of lead is generally only satisfactory for small amounts of lead (less than 0·10 g.), but recently a method has been described for the determination of as much as 1 g. of lead by deposition as lead dioxide from a perchloric acid/nitric acid solution. The value of the method is somewhat lessened by the fact that it is necessary to use an empirical factor after drying the deposit at 120° C.

The deposition of thallium as the metal on a platinum cathode is unsatisfactory, because the speed at which the metal is oxidised by atmospheric oxygen renders the weighing of the deposit inaccurate[18]. Besson[30], and Chretien and Longi[31] have shown that deposition of thallium as the oxide is also unsatisfactory, because of incomplete deposition from acid solution. Deposition of metallic thallium on an amalgamated electrode is useful for the separation of thallium, but is not very applicable to its determination, since the amalgam cannot readily be dried without loss of mercury[31,32]. Recently a reliable and accurate method has been described in which thallium is deposited on an anode as thallic oxide from an ammoniacal medium containing ammonium nitrate and cupric ions. The function of the copper is to act as a depolariser, preventing the deposition of metallic thallium on the cathode. Some of the copper unfortunately deposits with the thallic oxide so that a correction is necessary. This is done by solution of the thallic oxide in nitric acid and electrolysis of the solution for copper. Although normally it is necessary to have the thallium in the lower valency state, thallic hydroxide is not precipitated by ammonia in the presence of ammonium nitrate, so that the oxidation state of the thallium before electrolysis is of minor importance. The method cannot be recommended in the presence of large amounts of aluminium or iron because of the danger of occlusion of the thallic deposit on their hydroxides. A few ions, *viz.*, arsenic, chloride and phosphate interfere by inhibiting the precipitation of the oxide; and manganese, lead, bismuth and antimony (in excess of 3 mg.) interfere by contamination of the deposit.

The Determination of Bismuth

Electro-analytical methods for the determination of bismuth under constant current conditions are generally unsatisfactory because of the non-adherent nature of the metallic deposit on the cathode, and because of incomplete deposition. Deposition at controlled potential is more satisfactory. The addition of hydrazine sulphate to a sulphuric acid-nitric acid medium was found by Kny-Jones[33] to prevent anodic deposition of bismuth and to ensure good electrodeposition of the metal. Norwitz[34] used hydrazine sulphate in perchloric acid medium to effect a better determination, but normally silver, copper, lead, mercury, cadmium, tin, antimony and arsenic are co-deposited, so that a preliminary separation of bismuth is necessary. A more rapid method has since been evolved[35], however, in which the bismuth is precipitated from nitric acid containing peroxide and a trace of hydrochloric acid. The function of the peroxide is to oxidise nitrous acid which may be formed during solution of the sample in nitric acid, and the chloride ion appears to act in an analogous fashion to its use by Scherrer, Bell and Mogerman[36] in the determination of copper by deposition from nitric acid. A tendency for anodic deposition of bismuth was overcome by use of a spiral anode. Amongst the limitations of this new method are the facts that it cannot be used for bismuth in the presence of lead since the latter deposits at the anode as dioxide, inducing a similar deposition of bismuth, and that copper, antimony and silver contaminate the deposit. The maximum amount of bismuth determined was 0·35 g., using a current of 2 amps./sq. dm. for 10 minutes and 1 amp./sq. dm. for the remaining 50 minutes.

The Determination of Antimony and Tin

The electrodeposition of antimony from acid solutions is unsatisfactory because of the non-adherent nature of the deposit and incomplete deposition[37]. Deposition from sulphide solutions gives high results owing to the occlusion of oxygen and sulphur compounds[18,37,38]. Schoch and Brown [39] have described a satisfactory method for the deposition of antimony from hydrochloric acid, however, in which hydroxylamine hydrochloride was added, but careful control of temperature

and potential is necessary. The method has lately been modified[40], however, so that neither temperature nor potential are critical. This modification ensures that all the antimony is in the pentavalent state by oxidation with hydrogen peroxide before electrodeposition, and it is capable of determining as much as 0·4 g. of antimony. A preliminary separation was necessary in the presence of silver, copper, lead, cadmium, bismuth, tin, and arsenic.

Lindsey[41] has shown that losses previously noted by various authors in the recovery of tin from tin solutions are probably due to re-solution of the tin deposit during washing and disconnection, and has more recently[42] outlined a procedure for the quantitative deposition of tin from stannous and mixtures of stannous and stannic chloride solutions with an average error of ± 0·05 mg. on quantities of tin in the region 0·1–0·3 g. The procedure employed was the one previously described by Sand[43,44].

Determination of Silver and Gold

Norwitz[45] has commented on the fact that errors in the gravimetric determination of silver as silver chloride arise from the photochemical instability of the precipitate and the difficulty of removing the last traces of water from the silver chloride, and has suggested an electro-analytical finish which avoids both these sources of error. Alkali cyanides and ammonia were examined as solvent agents for the precipitate, but their action on the coagulated silver chloride was very slow, and solution was finally effected by dissolving both the filter paper and the precipitate in a mixture of sulphuric, perchloric and nitric acids. After dilution the silver was deposited by the cyanide method of Sand[19].

Several electro-analytical methods are available for the determination of gold, *viz.*, the cyanide method[18], the thiocyanate method[18,46], the sulphide method[18,47] and the hydrochloric acid method[19], but they are seldom used, because it is necessary to get rid of the nitric acid in which the gold is usually dissolved before these methods can be applied, and this constitutes a rather tedious process. Recently a method has been described which avoids evaporation[48] by neutralising the solution of gold in nitric and hydrochloric acids with sodium hydroxide and making alkaline so that the gold passes into the anionic salt of the alkali metal ($Na\ AuO_2$). Other heavy metals deposited simultaneously with the gold so that a preliminary separation was required. The presence of cyanide caused low recoveries.

Miscellaneous Determinations

A method for the electrolytic separation of rhodium and iridium has been proposed by MacNevin and Tuthill[49] in which the rhodium is deposited at controlled cathode potential. The co-deposition of iridium is prevented by oxidation to the tetravalent state, addition of ammonium chloride and destruction of complex ions. In the determination of rhodium by this method, an accuracy of ± 0·3% was claimed.

The use of mercury or amalgamated mercury cathodes has been examined for the determination of mercury, nickel, cobalt, iron and zinc by Böttger[50]. Electrolysis was effected in a medium of sulphuric acid with addition of hydrazine to prevent oxidation of the cathodic amalgam. Laisseur[51] has used electrodeposition by the graduated potential method for the direct separation of the main groups: silver and mercury; copper, bismuth, antimony and arsenic; lead and tin; cadmium; and lastly zinc, nickel and cobalt. Separation within the groups was effected by standard chemical methods.

Llacer[52] has developed a micro-deposition method for the determination of cobalt by controlled potential deposition from an electrolyte containing hydrazine as a depolariser. Haenny[53] and his co-workers have used a copper cathode for the quantitative deposition of radioactive iron Fe_{59}, while Norwitz[54] studied the deposition of iron from a phosphoric acid electrolyte. The electro-analytical determination of nickel and copper in plating bath solutions has been discussed recently by Silva Fangueiro[55], while Patterson and Banks[56] have developed the electro-analysis of zinc in zinc/thorium alloys containing more than 1% of zinc.

The most favourable conditions for the separation and successive determination of copper, bismuth, lead and tin from a tartrate medium by controlled potential deposition on a platinum cathode have been established by Lingane and Jones[57], and the importance of such factors as *p*H, temperature, tartrate concentration and cathode potential has been carefully outlined.

In conclusion, it is of interest to note that quantitative deposition of plutonium in amounts of as much as 0·1 g. has been described by Miller and Brouns[58]. The authors, however, were not concerned with the evolution of an analytical method.

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Use of Perchloric Acid in Removing Interferences Prior to the Electrolytic Determination of Lead as Lead Dioxide

By George Norwitz and Irwin Norwitz*

IT is well known that chloride, bromide, arsenic, antimony, tin, and reducing organic materials interfere with the deposition of lead as lead dioxide[1,2]. In this paper the authors describe how these interferences can be eliminated by fuming with perchloric acid, and then electrolysing the lead dioxide from a perchloric-nitric acid medium. Chloride and bromide are removed by simple fuming with perchloric acid. Arsenic, antimony and tin are removed by fuming with perchloric acid in the presence of hydrobromic acid. This removes these elements as the volatile bromides. The organic materials are destroyed by fuming with a mixture of perchloric and nitric acids.

Procedure

For Solutions Containing Chloride or Bromide

Add 15 ml. of perchloric acid to the solution and evaporate to strong fumes of perchloric acid. Dilute to 160 ml. with water and add 7 ml. of nitric acid and 20 ml. of copper nitrate solution (prepared by dissolving 11·6 g. of $Cu(NO_3)_2 \cdot 5H_2O$ in 500 ml. of water). Electrolyse for 1 hour at 2 amp./sq. dm. using platinum gauze cathodes and platinum gauze or sheet anodes. Immerse the anodes in water and in alchohol, dry at 110° C. for a half-hour, cool, and weigh the deposit as lead dioxide. The factor (empirical) for converting lead dioxide to lead is 0·864.

For Solutions Containing Arsenic

Add 15 ml. of perchloric acid and evaporate to a volume of about 30 ml. Add 20 ml. of hydrobromic acid and evaporate to fumes of perchloric acid with the cover lid ajar. Wash down the sides of the beaker and again evaporate to fumes of perchloric acid. Dilute with water, add nitric acid and copper nitrate solution and electrolyse the lead as described in the first procedure.

For Solutions Containing Antimony or Tin

Add 15 ml. of perchloric acid and evaporate to a volume of about 30 ml. Add 20 ml. of hydrobromic acid and evaporate to fumes of perchloric acid with the cover lid ajar. Add 10 ml. of water and 20 ml. of hydrobromic acid and again evaporate to fumes of perchloric acid. Wash down the sides of the beaker and evaporate to fumes of perchloric acid once more. Dilute with water, add nitric acid and copper nitrate solution and electrolyse the lead as described in the first procedure.

For Solutions Containing Organic·Materials

Add 15 ml. of perchloric acid and 25 ml. of nitric acid and evaporate to fumes of perchloric acid. Dilute with water, add nitric acid and copper nitrate solution and electrolyse the lead as described in the first procedure.

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TABLE I.—RESULTS FOR LEAD.

Interferences Present	Lead Present	Lead Found
	gram.	gram.
20 ml. of hydrochloric acid	0·0500	0·0502
20 ml. of hydrobromic acid	0·0500	0·0497
10 ml. of hydrochloric acid and 10 ml. of hydrobromic acid	0·0500	0·0496
10 ml. of hydrochloric acid and 10 ml. of hydrobromic acid	0·1000	0·1002
0·2 gram of arsenic	0·0500	0·0500
0·2 gram of antimony	0·0500	0·0496
0·2 gram of tin	0·0500	0·0498
0·15 gram of arsenic and 0·15 gram of antimony and 0·15 gram of tin	0·0500	0·0502
0·15 gram of arsenic and 0·15 gram of antimony and 0·15 gram of tin	0·1000	0·1002
3 grams of tartaric acid	0·0500	0·0498
3 grams of tartaric acid	0·1000	0·0998
3 grams of succinic acid	0·0500	0·0501
3 grams of succinic acid	0·1000	0·0999
2 grams of cupferron	0·0500	0·0501
Filter paper containing the lead as lead sulphide*	0·0500	0·0498
Filter paper containing the lead as lead sulphide*	0·1000	0·0998

* The lead sulphide was precipitated from a buffered formic acid medium and filtered.

Results

Results obtained by the authors using the above methods are shown in Table I. The lead was added in the form of a lead nitrate solution prepared by dissolving 2·5000 g. of pure sheet lead in 30 ml. of nitric acid (1 to 1) and diluting to 500 ml. in a volumetric flask.

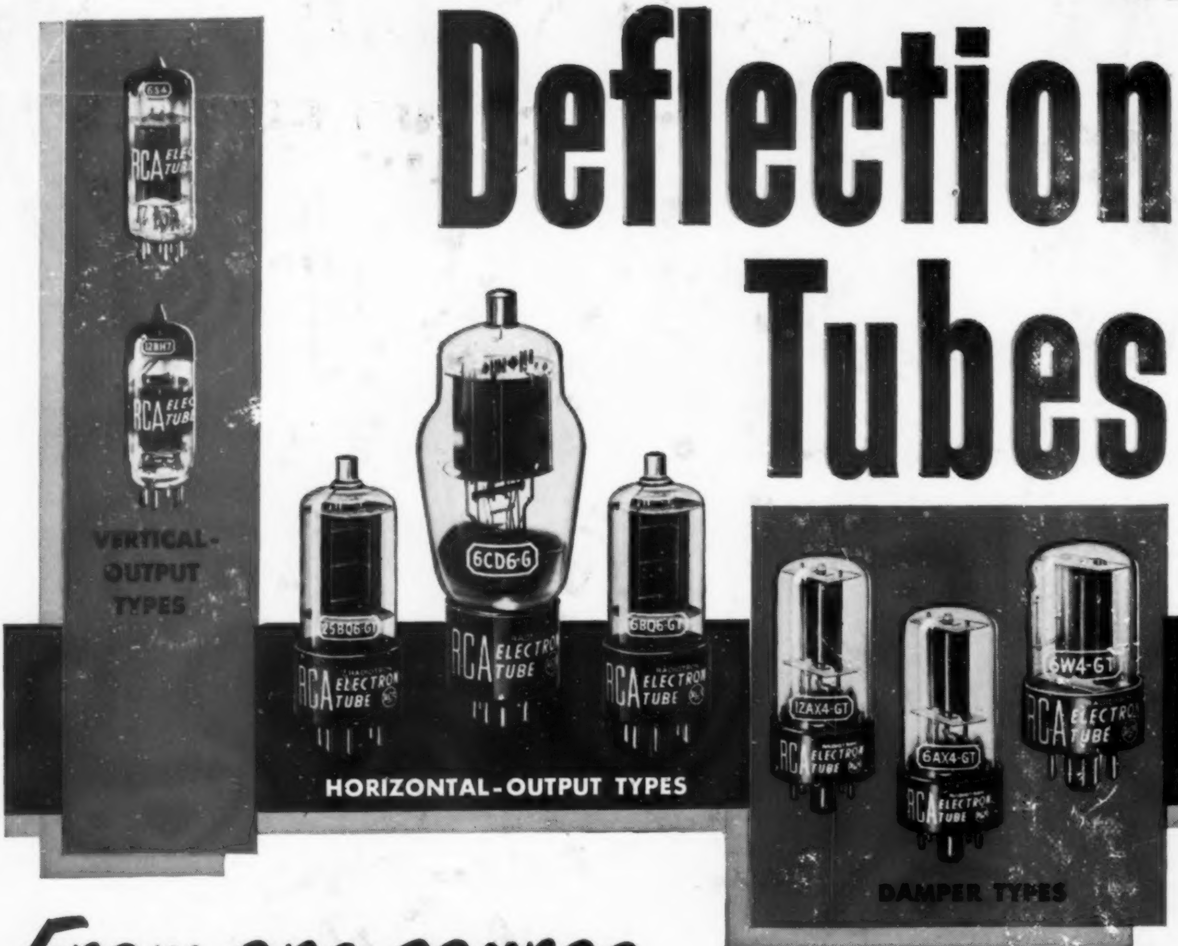
Discussion

If more than 0·5 g. of salts are present, there is danger of loss by spattering during the heating on the hot plate with hydrobromic acid in the removal of arsenic, antimony and tin. Therefore, if more than this amount of salts is present, evaporate off most of the hydrobromic acid by heating for four hours in the steam bath in an uncovered beaker and then evaporate off the residual hydrobromic acid by heating on the hot plate.

The destruction of the organic materials listed in Table I (tartaric acid, succinic acid, cupferron and filter paper) by the use of a mixture of perchloric and nitric acids is perfectly safe. However, the authors do not know with certainty whether the procedure could be applied to the destruction of other organic materials that might interfere with the deposition of lead dioxide.

Unsuccessful attempts were made by the authors to apply the methods described in this paper to the removal of other substances that interfere with the deposition of lead dioxide, namely iodide, selenium, tellurium, and mercury. It was found that interference from iodide was not eliminated by fuming with perchloric acid, very poor deposition of the lead dioxide being obtained. Fuming with the perchloride acid probably converts the iodide to some non-volatile acid such as iodic acid that interferes with the deposition. According to the experiments of the authors the removal of selenium, tellurium and mercury by the use of the hydrobromic acid treatment was incomplete.

Deflection Tubes



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